INSTITUTE NOTES.

MARCH 1939.

FORTHCOMING MEETINGS.

Thursday, 20th April, 1939, at 5.30 p.m. at the Royal Society of Arts, John Street, London, W.C. 2. **Annual General Meeting.**

SUMMER MEETING.

The Summer Meeting of the Institute will be held at Birmingham from May 22nd–24th, 1939, under the Presidency of Professor A. W. Nash, M.Sc., M.I.Mech.E. The objects of the meeting are to review and interpret recent work on fuels and lubricants for use in internal combustion engines.

The programme of the meeting, together with details regarding Ladies' Visits, Registration, etc., are given in the circular sent out separately to members.

A Summary of the programme is given below :

Monday, May 22nd.

Evening. Informal Reception by the President at the Grand Hotel, Birmingham.

Tuesday, May 23rd.

Morning. Technical Session.—Knock-Rating.

Afternoon. Technical Session.—Lubrication.

Evening. Reception in the Grand Hall, The University, Edgbaston.

Wednesday, May 24th.

Morning. Technical Session.—Fuels for Compression-Ignition Engines ; Lubrication.

Afternoon. Visits to Austin Motor Company and Morris Commercial Cars Ltd.

Evening. Dinner and Dance at the Grand Hotel.

STUDENTS' SECTION (LONDON BRANCH).

Tuesday, 4th April, 1939, at 6.15 p.m. at the Sir John Cass Technical Institute, Jewry Street, Aldgate, London, E.C. 3. Annual Open Meeting. **"High Speed Engines,"** by H. R. Ricardo, F.R.S.

Wednesday, 19th April, 1939, at 5.45 p.m. at the Offices of the Institute, The Adelphi, London, W.C. 2. **"The Oilfields of Iraq,"** by N. Pachachi.

TRANSFERS TO NEW CLASSES OF MEMBERSHIP.

The Temporary Regulations relating to the transfer of the existing members to the new classes of membership, as set out in the leaflet sent to all members of the Institute, were approved at the Special General Meeting held on 10th January, 1939.

Members who wish to transfer to one of the new classes of membership are requested to submit their applications as early as possible on the forms provided for the purpose at the back of the leaflet convening the Special General Meeting.

STUDENTS' MEDAL AND PRIZE.

The Council has decided that the Students' Medal and Prize in 1939 will be awarded for a thesis on a set subject, and not for theses on subjects chosen by the candidates themselves.

A short list of alternative subjects on which theses are invited will be issued by the Council to all Students of the Institute after 30th June, 1939.

CANDIDATES FOR ADMISSION.

The following have applied for admission to the Institute or transfer to another grade of membership, and in accordance with the By-laws the proposals will not be considered until the lapse of at least one month subsequent to the issue of this *Journal*, during which any Member or Associate Member may communicate by letter to the Secretary, for the confidential information of the Council, any particulars he may possess respecting the qualifications or suitability of any candidate.

The object of this information is to assist the Council in grading candidates according to the class of membership.

The names of the candidate's proposer and seconder are given in parentheses.

ARMSTRONG, Valentine, Student, 27, Kensington Gardens Square, London, W. 2. (*A. C. Egerton ; V. C. Illing.*)

BRADDICK, Herbert James William, Engineer (*Anglo-Iranian Oil Co., Ltd.*), 25, Pickhurst Mead, Hayes, Kent. (*A. C. Hartley ; A. E. Dunstan.*) (*Transfer to A. M.*)

CLIFFORD, Joseph, Chemist, 5, Arlington Avenue, South Shore, Blackpool. (*W. H. Cadman ; C. W. Wood.*)

CRIGHTON, Robert Mill McCombe, Engineer (*William Briggs & Sons, Ltd.*), 30, Thomson Street, Dundee, Angus. (*A. D. McLuckie ; W. F. Murray.*)

CROSSLEY, Stanley, Director (*British Transformer Oil & Lubricants, Ltd.*), "Southview," Old Forge Close, Stanmore, Middx. (*E. J. Dunstan ; B. J. Vavasour.*)

FRAME, Alexander, Works Manager (*Roman Bank Crude Oil Works*), Woodside, Drumshoreland, Broxburn, W. Lothian. (*R. Crichton ; G. H. Smith.*)

HALL, John Desmond, Chemical Engineer (*Foster Wheeler, Ltd.*), "Earlscroft," 82, Cecil Park, Pinner, Middx. (*A. W. Nash ; R. K. Fischer.*) (*Transfer to A. M.*)

HATT, Ernest Richard, Works Manager (*Dussek Bitumen & Taroleum Ltd.*), "Kingarth," Longdon Wood, Keston, Kent. (*F. H. Garner ; A. Osborn.*)

HILTON, John Sydney, Chemist (*Belgrave Oil & Grease Co. (Leeds), Ltd.*), "Belgrave," 19, Sandhill Oval, Alwoodley, Leeds. (*F. Dakin.*)

LEWIS, Philip Cottrell, Mechanical Engineer, The Whessoe Foundry & Engineering Co., Ltd., Darlington, Co. Durham. (*E. R. Cartwright ; H. D. Demoulins.*)

LUMB, Erling, Chemist (*Munster, Simms & Co., Ltd.*), "Larvik," 71, Rugby Avenue, Bangor, Co. Down. (*L. R. Phillips ; A. W. Nash.*)

MACARTHUR, Hector, Chemist (*W. B. Dick & Co.*), 38, Riverside Road, Newlands, Glasgow, S. 3. (*W. M. Cumming ; G. H. Smith.*)

MARTIN, Bernard Davis, Director (*Germ Lubricants, Ltd.*), City Gate House, Finsbury Square, London, E.C. 2. (*J. E. Southcombe.*)

MARTIN, Jack William, Engineer (*Whessoe Foundry & Engineering Co., Ltd.*), Greengates, Caledon Road, Beaconsfield, Bucks. (*E. R. Cartwright ; H. D. Demoulins.*)

METCALFE, Thomas John, Chemist (*E. Joy & Sons, Ltd.*), 3, Grafton Villas, Stanks, Leeds. (*G. V. Davies ; J. R. Smellie.*)

MORTIMER, George Abbott, Chemist (*Pumpherston Shale Oil Co.*), 47, Queens Avenue, Blackhall, Edinburgh. (*R. Crichton ; G. H. Smith.*)

NAKIB, Mohammad Abdul Ghazi, Engineer, Government Office Oil Measurement Section, Kirkuk, Iraq. (*A. W. Nash ; L. V. W. Clark.*) (*Transfer to A. M.*)

NEWEY, Clifford Samuel, Engineer (*Caribbean Petroleum Co., Ltd.*), 49, Dartmouth Street, West Bromwich. (*A. W. Nash ; L. V. W. Clark.*) (*Transfer to A. M.*)

NEWTON, Ernest John, Chemist (*Ernest Newton & Co.*), 353, Birmingham Road, Wylde Green, Sutton Coldfield. (*H. C. Tett ; A. Hamilton.*)

O'BRIEN, Kevin, Director (*H. F. O'Brien & Co., Ltd.*), "Thomond," Brookfield Avenue, Timperley. (*D. W. O'Brien ; J. Barrett.*)

O'MEARA, Terence Barry, Chemist, c/o Colas Products, Ltd., Norman House, Strand, W.C. 2. (*L. G. Gabriel ; J. S. Jackson.*)

PROCTER, Reginald Henry, Engineer (*English Drilling Equipment Co.*), 65, Elgar Avenue, Tolworth, Surrey. (*F. E. Cherry ; A. J. Vokes.*) (*Transfer to A. M.*)

RIGDEN, Peter Montgomery, Chemist (*Wailes Dove Bitumastic, Ltd.*), 12, Victoria Square, Jesmond, Newcastle-on-Tyne. (*R. Shaw ; H. C. Rampton.*) (*Transfer to A. M.*)

SAIB, Mohammed Ali, Engineer, c/o Ministry of Economics & Communications, Baghdad, Iraq. (*A. W. Nash ; L. V. W. Clark.*) (*Transfer to A. M.*)

TAYLOR, Theo Mallinson, Chemist (*Shell Marketing Co.*), 25, Matheson Road, West Kensington, W. 14. (*J. S. Jackson ; S. R. Hills.*)

WHITE, Colin McLuckie, Chemist (*Pumpherston Oil Co.*), 60, Main Street, Winchburgh, W. Lothian (*R. Crichton ; G. H. Smith.*)

WILD, Eric Herbert, Chemist (*Anglo-American Oil Co., Ltd.*), 9, Rusland Road, Wealdstone, Harrow. (*H. C. Tett ; E. B. Evans.*)

ARTHUR W. EASTLAKE,
Honorary Secretary.

JOURNALS WANTED TO PURCHASE.

The Institute is prepared to purchase copies of the following issues of the Journal at the price of 4*s.* 0*d.* each.

No. 152, June 1936
„ 171, January 1938
„ 174, April 1938

Journals should be forwarded to the Secretary, The Institute of Petroleum, The Adelphi, London, W.C.2. Only copies in good condition will be considered for purchase.

PERSONAL NOTES.

Mr. M. ALLAM has returned to Egypt after a long visit to the Hedjaz.

Mr. B. D. CAUTHERY is home from Iran.

Mr. E. K. DYKES has left for Trinidad.

Mr. J. C. JEWELL has returned from Iran.

Mr. H. H. MARTIN is home from Ecuador.

Mr. C. A. SANSOM is home from Burma.

Correspondence or *Journals* forwarded to the following members have been returned, and the Secretary would be pleased to receive any information regarding their present address: E. C. BROWN, K. BURTON, M. CAPPER, O. C. ELVINS, V. C. S. GEORGESCU, J. J. L. HAMILTON, J. R. HORTH, A. D. JONES, J. LANDER, H. R. LOVELY, I. LUSTY, F. MACKLEY, A. MACLEAN, G. P. MELVILLE, C. A. MOON, S. NICOL, S. PAPP, R. G. REID, N. D. ROTHON, H. G. SPEARPOINT, and A. H. WILLIAMS.

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VOL. 25. No. 185. MARCH 1939.

TANK STRAPPING.*

By P. KERR, M.A., B.Sc., A.I.C. (Member).

SYNOPSIS.

The paper suggests decision in advance on the maximum uncertainty permissible in the tank table which is to be produced. Measurements on the tank may then be made with the lowest permissible precision, and therefore with the greatest ease. Only those corrections and allowances which are necessary to secure the desired accuracy in the final table need be included.

Applying this principle, the paper sets out the chief precautions necessary when tables of high accuracy are to be prepared from external measurements of tank circumferences. Other methods of tank calibration are discussed only so far as they bear on the method considered.

The chief corrections to apparent external circumferences required to obtain the mean horizontal internal cross-sectional area for each course are then discussed. These include the effects of the temperature, tension or errors of graduation of the tape, and of displacements of the tape from the tank surface by vertical seam edges or other obstacles, or through local distortion of the tank plates. The effects of slight conicity and ellipticity of the courses are considered, as also that of tilt of the tank as a whole. Corrections depending on the mean temperature of use of the tank and its expansion under the head of oil in it are discussed, as also those for plate and paint thickness, and for internal fittings. The adjustment of tables for irregularities in the shape of the tank bottom is also considered briefly.

TANKS may be satisfactorily calibrated in many different ways. The method discussed below is that of " strapping " or measuring apparent circumferences outside the tank. Correction is made later for all factors which affect the final tables.

With this method the principal measurements may be taken while the tank is in use, and less important measurements may usually be taken from the drawings. In a busy installation it is seldom easy to empty a tank completely. It is, however, difficult to measure the effect of corrosion in the plates of old tanks without this.

PRELIMINARIES.

Before beginning work, the degree of accuracy required in the final tables should be decided. This decision avoids waste of time in taking unnecessarily exact measurements, and again secures inclusion of all precautions in working and corrections really required. If oil shortages or discrepancies require investigation later, it is essential to know how far one can rely on the tank tables.

This degree of accuracy is most simply expressed as a tolerance—say, the percentage error permissible in the final tables. The exact error present in tables is never known : if it were, the tables would be corrected. There is therefore some advantage in expressing the degree of accuracy aimed at or achieved as a " probable error " or " standard deviation."

Many non-technical considerations bear on the choice of such tolerances. Time and staff may be limited, and costs have usually to be kept down. A highly valuable product would call for smaller tolerances than would

* Paper presented for Discussion at the One Hundred and Eighty-fifth General Meeting of the Institute of Petroleum held on 10th January, 1939.

water. On the other hand, if a tank table is to remain in use for several years, it may pay to avoid even small errors. The total effect of these might ultimately add up to a considerable cash value, even with a relatively cheap product.

It is sometimes argued that since it is impracticable to measure the amount of oil in a tank exactly, and since the uncertainty of a single measurement may represent a large number of gallons, it is therefore unnecessary to aim at high precision in tank tables. In the writer's view, this is incorrect. First, the unavoidable errors in measuring oil quantities are often overestimated. Again, if gauging is properly carried out, the measurements will sometimes slightly over-estimate, sometimes under-estimate, the oil quantity. Taken over a sufficient series of measurements, such fortuitous errors tend to cancel out in percentage. If the tank tables contain any given percentage inaccuracy, this error will be present in all quantity calculations. The error is systematic, and does not tend to cancel out in percentage, no matter how many consecutive measurements are averaged. The utility of good tank tables in controlling working losses is obvious.

Technical considerations also bear on the selection of tolerances. Measurement difficulties increase rapidly as the error allowable is reduced. The precision with which measurements must be taken increases. Corrections negligible for rougher work must be included in finer calibrations.

To a great extent, therefore, details must depend on the circumstances of the case. To be of general use, this note necessarily describes methods of high accuracy. Those who feel that too many corrections and precautions are included are reminded that by choosing lower standards of accuracy, many of these may be neglected.

Calibration by Filling.

For a given amount of care, strapping tends to give higher percentage uncertainties on small tanks than on large. A common method of calibrating smaller tanks, often applied also to calibrate irregular parts of larger tanks, is to fill measured quantities of liquid into the tank, the resulting depth of liquid being measured after each addition. For any previously chosen degree of uncertainty in the results, the trouble and expense of this method by filling increase with the size of the tank calibrated. It is usual, therefore, to strap larger tanks and fill smaller. As the size of the tank decreases, a point is reached at which the cost of securing a given accuracy by strapping becomes greater than that by filling. It is at this point—admittedly somewhat indefinite—that one should change over from the one method to the other. This note does not fully discuss methods other than that of strapping, and this implies that tanks of suitably large diameter are alone considered. These normally approximate to vertical cylinders, and other shapes are not considered here.

Calibration from Drawings.

Tanks are now usually made in standard sizes, and the accuracy of their construction is much greater than formerly. The plates for any one course are usually interchangeable. Such tanks approximate very closely to the

dimensions shown on their drawings. Tables of very satisfactory accuracy may then be calculated from the drawings in advance of erection, this allowing the tank to be brought into use without delay.

Such tables do not, and cannot, allow for irregular deformation of the tank bottom on its foundations, but this part of a tank is seldom used for precise measurements. Oil received into or delivered from a tank may be fairly satisfactorily measured without bottom calibration, provided that the tank is not emptied. It is perhaps the rule rather than the exception to neglect such irregularities. Oil-stock figures are affected by this neglect. Where these are important, the tank bottom may be calibrated. In some cases water bottoms are put into tanks, which eliminates the effect on stocks, but storage space is wasted by this if the tank bottom is known to be tight.

Official regulations in many countries demand the production of tank tables from measurements made on the tank after erection. Except for this, in the author's view the production of the final tables for standard tanks by calculation from the drawings might usefully be applied more widely than at present. The verifications necessary are perhaps most open to those who purchase a large number of tanks. Comparison of recorded measurements on tanks previously erected with the corresponding figures calculated from the drawings, then shows whether the standard tanks of a particular maker match the drawings sufficiently closely to allow tables to be tentatively calculated as suggested.

Before tables so calculated are accepted as final for a further tank, the erection engineer should report for this that little or no reaming of the rivet holes has been necessary before rivets were driven, and also that the tank after erection showed little or no leakage at the seams. A single circumference may be measured at some convenient height on the tank as a check that all is in order. Such other check measurements as are easily made may also be taken. The savings are considerable, for not only are measurements largely avoided, but the standard table calculated for any particular tank may be used again for other tanks of the same size, so that calculation and printing are also reduced.

Even when direct measurements must be taken, the tank drawings, if available, give a valuable check. It is useful to calculate out beforehand the circumferences and other measurements which should be found by the strapper. Any notable deviation of his measurements from those expected then directs attention at once to the need for careful verification of the measurements found. Especially if the crew has to be sent some distance for the work, or if the production of reliable tables is an urgent matter, the saving as compared with later detection and verification of the discrepancies is often considerable.

In these preliminary calculations it is useful to give not only the figures expected, but also the tolerance allowable on each of these while still maintaining the required accuracy in the final tables. Rechecking of minor discrepancies in observed measurements is thus obviated.

Recalibration.

Decision to recalibrate tankage is usually taken only when discrepancies in measurements have become troublesome. Before this is decided on, it

is well to verify local measurement procedure on all the simpler points in which faults are likely to be found. If visual inspection of a tank shows it to be well made and free from flats or deformations, measurement of a single circumference at some convenient level, usually near the bottom of the second course, taken in conjunction with external measurement of the plate thicknesses, overlaps and heights for all courses, is usually sufficient to show whether the cost of recalibration is or is not justified. The course heights used in calculating the tank capacities must naturally be the internal heights, but one can usually estimate these with sufficient accuracy from the external course heights and the seam overlaps. Much depends on the condition of the tank, but for tanks without visible irregularities an excellent check is often obtained.

Further check on the accuracy with which the final tables have been constructed is often obtainable from oil transfers. The measurements made in another tank from which the newly calibrated tank is filled, or into which it is emptied, are compared with those taken in the tank under check. Auxiliary tanks used for this must themselves be known to be well calibrated. The precision with which such measurements, purely internal to the installation, are made in ordinary routine is sometimes not very high. It may be necessary to average a sufficiently long series of transfers before a good comparison is obtained. The greater the variation in percentage difference between separate comparisons, the greater the number of individual cases which must be averaged before the comparison is relied on.

This comparison is affected by pumping losses in the case of volatile products. Some compensation for this evaporation may be obtained by taking comparisons both into and out of the tank concerned. If the pumping operations are reasonably similar, then the shortage due to loss in receipts will be balanced, at least to some extent, by the shortage due to loss in deliveries, both losses being best expressed as percentages on the quantities handled.

Strapping Levels.

The upper edges of external courses form a very convenient ledge on which to rest the tape if a circumference at the bottom of the next higher inner course is being measured. In the author's view, the temptation to use this as a level at which to measure a circumference should be resisted. Rivets are driven hot, and unless the plate is very thick, it is often possible to detect a local spreading of the plate. This local distortion is usually completely negligible in its real effect on the tank capacity. If the tank is strapped at this level, however, the distorted circumference is then taken as applying to the whole height of the course, or even to more than one course, according to the number of levels measured. The edges of plates are the parts most liable to slight irregularities. It is preferable to avoid these levels as much as possible in strapping.

If the tank to be strapped has been painted, a little paint often drains down and is caught by the upper edges of outer plates. The effect is irregular, and a satisfactory circumference can often be obtained at this level only after removal of the paint. The trouble is best avoided by making the rule of not strapping within, say, four inches of the seam. This rule is also applied when strapping lower circumferences on outer courses.

"Referee" methods for strapping tanks usually call for at least one circumference to be measured on each course. If there is sufficiently close agreement between the observed measurements and those calculated from the drawings, then some of the courses—say, every second—might reasonably be omitted from measurement. Appropriate rules for such omissions are, however, a little difficult to formulate in a manner suitable for use by the strappers. It may also be doubted whether such rules would effect substantial economies. Once the expense of getting the strapper's crew and gear to the tank has been faced, the additional cost of strapping a few more circumferences is not very great. The saving might be useful in the case of a very busy crew. Those methods in which a smaller number of courses to be strapped is laid down in advance of any information as to the regularity of the tank, leave one somewhat uncertain as to the real accuracy of the resulting tables, if not of the results themselves.

If this view is accepted, then a circumference is measured near the bottom of each course of the tank, with another near the top of the top course. These circumferences should not be measured too close to the joints, but should be taken, say, four to six inches above horizontal seams. The exact levels used are chosen so that the tape passes with a minimum amount of interference between the rivets of the vertical seams.

Strapping Tapes.

Circumferences are most conveniently measured by means of a steel tape. This is stretched around the tank at some convenient tension—usually 10 lbs. It is a little difficult to be quite sure that the tension is transmitted uniformly around the tank. Possible errors resulting from this would be minimized if the tape were of large cross-sectional area. This would be secured by using a wide or thick tape.

Against this, a narrow tape is to be preferred if it is to pass among the rivet-heads at the vertical seams with the minimum amount of interference. It is not desirable that the tape should be so wide that it must pass entirely over these rivet-heads. Correction of circumferences so measured for the displacement of the tape by the rivet-heads would not be difficult, but the great practical advantage of having the tape pass nearer the tank surface is that the rivet-heads help greatly in keeping the tape to its proper path round the tank.

Again, tapes are most conveniently kept wound up on a frame when not in use. If the tape is thick, it tends when unwound to retain the set given it by this winding, which leads to great practical difficulties.

Both the width and the thickness of the tape are therefore a matter of compromise. It is desirable that the tape should be flat, since it then tends naturally to follow its proper path between the supporting rivet-heads.

The methods used by reputable makers in their manufacture of steel tapes are of very high precision. Errors of graduation in these seldom amount to more than a few parts per 100,000 of the length measured. Tapes used in tank strapping should of course be calibrated stretched horizontally at the tension at which they are to be used. They vary in length with their temperature. The temperature at which the small corrections occasionally

found necessary are given is now always 68° F. (20° C.). If errors are present in the tape itself, the apparent circumference figure is corrected for their effect, the corrections being given on the calibration certificate.

Tape Temperature.

Since it is difficult to measure the tape temperature, and since this affects the readings obtained, it is desirable, when good figures are required, to strap tanks at such a time of day that the air and the tank contents are at about the same temperature. Especially if the plate surfaces are rusty, pitted or painted, the tape may easily not be in good thermal contact with the tank. In strong sunshine the high temperature taken up by the tape in such cases is readily detectable by running the finger along it. Another method for avoiding the uncertainties which arise from this cause is to use a nickel alloy tape of very low coefficient of thermal expansion. Such tapes are, however, rather expensive. Since they are brittle, such care must be taken in their use that, in the writer's opinion, they are hardly practicable for routine strapping.

If their temperature changes, steel tanks expand or contract both horizontally and vertically, but such changes in their dimensions are not measurable with steel tapes, since these rapidly take up the temperature of the materials with which they are in contact. The effect of this on tank tables is perhaps rather unexpected. If a tank is strapped when the air, the tape and the tank plates are all at 20° C., then, setting aside other corrections possibly necessary, the circumference measured will be correctly that of the tank at 20° C., if this is the temperature at which the tape is correct. If the air, tape and tank temperatures are all raised—say, to 30° C.—then the steel tape will expand at closely the same rate as the steel tank. The circumference obtained will not be the true circumference of the tank at 30° C., but will still be that at 20° C., the temperature of calibration of the tape. If the precautions described in the preceding paragraph have been taken, the circumferences obtained may and must be regarded as those which the tank would have at the temperature of calibration of the tape.

It is then permissible and possible to correct the tables to the normal or average working temperature of the tank. If the tank has been strapped without these precautions, a small error, the limit of which is not exactly known, will be introduced.

Tape Displacements.

The courses of tanks, especially those of in-and-out construction, approximate closely to right cylinders on the same axis, each course differing slightly in its dimensions from those above and below it. If the tape, in strapping, is made to take a path lying in a plane at right angles to the common axis, the circumference so measured will be approximately circular. The exposed plate edges define such planes, even if the tank is tilted. The path which the tape should take may either be marked in advance by measurement from the nearest convenient edge, or the tape may be adjusted to the path after it has been placed in approximate position.

An assumption underlying the method is that the circumferences so measured are sufficiently circular to be corrected to true circles, and that the cross-sectional area of the tank at that level may be calculated from these. If the tank were truly circular, as might be the case with a welded tank, then the tape would lie in contact with the plate surface throughout its whole length. Any departure of the tape from the tank surface is therefore a warning that steps may have to be taken to apply an appropriate correction.

If the tank plates have been badly handled before erection, or if the tank sides have been blown in by the wind during this, permanent flats or deformations may be left in the tank sides. Slight flattening is also usually noticeable above and below vertical seams, especially with half-plate staggering of these between courses. The formation of a flat generally forces the plate on each side of the flat to project a little outside the circumference of an undamaged plate. Relatively slight flattening is therefore sufficient to cause the tape to leave the tank surface. The strapper should be required to verify that the tape maintains normal contact with the tank over each circumference measured.

When the flattening is severe and a considerable length of tape comes out of contact, the levels affected must be considered as irregular. The method of filling might be applied to obtain the table for the levels affected, or the effect of the deformation on the tank capacity may be obtained by suitable survey. Such cases are relatively rare, since serious deformation of the plate will generally result in erection difficulties. The question is discussed more fully below.

The tape may leave the tank surface through passing over rivet-heads. The level strapped may usually be chosen to avoid these supporting internal fittings, but it is often impracticable to avoid those of doubly or trebly riveted vertical seams. Even with single riveting, vertical seam edges normally displace the tape. Butt-straps, and in some cases external fittings, cannot be avoided.

Step-Overs.

All these cases are met by using the " step-over," a rigid frame supporting two scribing points. Its shape and the gap between the points are so chosen that when one of these is applied to the tank surface on one side of the obstacle, the other may be brought in contact on the other side. The points should touch the plates well clear of any local distortions. Convenience may demand more than one size of step-over.

For large obstacles, such as fittings, the level strapped is first marked on both sides of the obstacle. A fine vertical line is then drawn at right angles to this tape path on one side, and the partial circumference up to that line is measured with the tape. One of the scribing points is then placed on this " zero " line, and the other point made to scribe a second zero line at the proper level on the other side. Strapping is continued on from this second line, the effective length of the step-over gap being added in later to the parts of the circumference measured with the tape.

This effective length is not, of course, the linear distance between the scribing points, but the arc of the undisturbed course circumference contained between them. This varies with the radius of the course, most

rapidly if the tank is of small diameter. If the linear distance between the points is measured, it is easy to calculate the effective length for any course radius. If preferred, the value of the gap may be determined on the course itself. For this, the tape is stretched out on the course exactly as if a circumference were being measured, though it is not necessary that the tape should encircle the tank completely. The step-over is then applied to the tape where it lies in good contact with the tank surface—that is, usually about the middle of a plate. The distance read off on the tape between the scribing points is then the effective length of the gap for that course.

It is best to repeat this measurement at several points around the tank circumference, and to take the average of the results as the value for the gap. This is esssential if the course is out of round—say, elliptical—in section.

Alternatively, the circumference may be strapped over the obstacles, as if they were not there. This is the more convenient method for a series of small similar obstructions—say, vertical seams and their rivets, or butt-straps. With the tape still in position under tension after strapping, the step-over is applied across the obstacles, and the apparent distance between the points is read off on the tape. The true value of the gap for the particular circumference being known, the difference between this and the length read off on the tape is the effect of the obstacle on the circumference measured.

The tape will normally pass in the same way through all vertical seam or butt-strap rivet-heads on any one course. The effect of one obstacle will then be the same as that of another of the same type. It is then unnecessary to apply the step-over across each obstacle. A sufficient number of corrections are obtained to verify that the obstacles are really similar in effect, and to give a fair average figure for the correction. In correcting the circumference, this average is multiplied by the known number of obstacles. Since the average is to be multiplied, often by a considerable number, great care should be taken in the individual observations. The average may usefully be calculated a decimal place further than is obtainable with the individual measurements, to avoid arithmetical error from the subsequent multiplication.

Butt-strap corrections naturally vary with the size and thickness of the butt-strap. Even for vertical seam corrections, the average correction for one course should not be applied to correct circumferences on another course, unless the courses are similar and the tape passes in the same way through the rivet-heads.

When the types of tank calibrated are standard, it is possible to tabulate adequate corrections, best by averaging previous measurements made on similar tanks. This obviates much of the need for the step-over, especially on the higher parts of the tank, which are usually troublesome to reach. The width of the tape used in strapping must also have been standardized before such tables can be applied, since in general this width will affect the amount by which the tape is raised from the tank surface in passing through vertical seam or butt-strap rivet heads.

On tanks which are not standard, or not to the same standard, tape paths across vertical seams may vary, altering the value of the correction applicable. The edge of a seam normally projects above the cylindrical surface of

the tank, but seams sufficiently re-entrant to allow the tape to pass over them clear of all interference are occasionally met with.

Oval and Conical Courses.

Without causing the tape to leave the tank surface, tank courses may depart from cylinders either horizontally or vertically. They may be elliptical in section, or otherwise out of round. These general deformations must be severe before they have any substantial effect on the tables for the tank. The circle is the figure of maximum area for a given circumference. If therefore a course, originally circular in section, is slightly deformed in such a way that it still gives the same measured circumference, the resulting alteration to the cross-sectional area is necessarily of at least the second order. If, for example, a course originally circular is slightly deformed into an ellipse of the same circumference, the percentage correction to the calculated cross-sectional area of the tank is approximately $9e^4$, where e is the eccentricity of the ellipse formed. The tank bottom and the heavy plates of lower courses tend to keep these levels circular, as also roof-girders the top courses. Tanks are most often out of round about mid-height.

What has just been said applies also to local deformation of plates. Slight local flattening of a plate throws its centre towards, and its borders outwards from the tank centre. The real perimeter of the tank is not altered, and its cross-section at that level only negligibly. If the tape still maintained contact with the tank surface, there would seldom be any need to correct the tables. The need for correction usually arises from the tape's leaving the tank surface, which, so to speak, gives an erroneous circumference.

If a circumference on each course is called for, the flat may often be avoided by choosing a different level at which to strap, relying on the proof just given that slight flats do not appreciably affect capacities. Special survey of the flattened area, or the use of the method of filling, is seldom really justified.

If tanks are strapped when they are full of oil or nearly so, the oil head tends to round out irregularities. As has been shown, this does not usually alter the tank capacity, but it does reduce the risk of troubles from local flattening, so that it is advantageous to strap tanks when they are full.

"Shingled" tanks, or those in which the lower edge of each course lies outside the upper edge of the course below, are sometimes met with. Such tanks may be telescopic in construction, or the general diameter may be preserved by coning the courses.

If the course is only slightly coned, then a circumference might be measured at the middle of the courses. This would then be sufficiently close to the mean circumference of its course to be used in calculating the tank table. The author prefers, however, to retain the method suggested above—that of measuring a circumference near the bottom of each course, with one more near the tank top. The circumference measured at the bottom of any course is then greater than the proper average for the course. A sufficiently correct average for the course is, however, obtained by calculating a circumference for the top of the course, with subsequent averaging of this with the measured bottom circumference.

Calculation of the required top circumference is usually not difficult. The bottom circumference of the next higher plate is available. The chief cause of difference between this and the calculated circumference required is the thickness of the overlap between the two courses. If therefore the measured circumference on the next higher plate is corrected for the effect of its plate thickness, the required second circumference is obtained. The labour consists in taking the appropriate correction from the tabulated effects of plate thickness mentioned below, subtraction of this from the upper plate circumference, with subsequent averaging as described.

Neither of the two procedures just discussed is theoretically quite correct. In forming the table, it is the true capacity of the course that is to be aimed at. The volume of the frustum of a cone is not obtained exactly by multiplying the area of its mid-section by the height of the frustum. The correction is usually entirely negligible in the cases under consideration, but the method suggested allows a small and easily calculated correction to be applied, if this is thought necessary. The chief advantage of the suggested method, however, is that it allows a single standard strapping procedure to be adopted for any common form of tank construction.

Where coning is intentional and severe, radically different methods of calibration would be adopted. Such cases arise with coned bottoms of agitators, and the like.

Expansion under Oil Head.

When tanks are strapped full of oil, slight conicity of their courses is always present. The metal of the plates expands under the oil head, and this effect is greater at the bottom of a course than at the top. The procedure just given is sufficient to allow for this conicity, except sometimes for the bottom course.

The bottom plates stiffen the resistance to expansion of the lower part of the bottom course. It is usually impossible to detect any expansion under oil head near the bottom angle iron. Higher up the course the resistance lessens, until at the top, if not before, the expansion under oil head is very closely that calculated from theory. The shape of the bottom course is then roughly that of an inverted conical frustum, but the sides of this frustum are slightly convex outwards. To allow properly for this effect in the tables, it may be necessary to measure a circumference near the middle of the course, in addition to that near the bottom. The bottom course is then treated in calculation as if it were two courses, each of slight conicity.

Tilt.

If a tank is tilted from the vertical, then the oil surface is not circular, but elliptical. The capacity of the tank per unit depth, gauged in the usual way, is greater than if the tank were truly vertical. In almost all practical cases the effect is so slight that it may be neglected. If a tilted tank were brought back to the vertical and then inclined to the other side, the surface area of the oil in it would pass through a minimum as the tank passes through the vertical. The effects of slight tilt are, therefore, of the second order. It may easily be shown that a tank must be tilted by 0·8

of a degree from the vertical before its volume per unit depth, taken vertically, is altered by 0·01 per cent. Long before such tilts are reached—say, by settlement of the tank foundations—the tank has usually been re-levelled by the engineers in charge. Especially if the tilted tank stands among others which are vertical, the eye appears to be very sensitive to any lack of verticality. The strapper may be asked to report any visible tilt, but this usually leads to more cases being reported than really require correction.

Plate and Paint Thickness.

The capacity of a tank is determined by its internal, not its external dimensions, and the latter must therefore be suitably corrected. The thickness of the plates and of the paint, if any, over which strappings have been taken must be allowed for. One may, if one chooses, calculate the external radius of each course, and subtract the corresponding plate and paint thicknesses. It is, however, mathematically equivalent, and in practice more convenient, to reduce external circumferences directly to internal by suitable allowances for plate and paint thicknesses.

These two thicknesses are conveniently taken together. For every 1/16th of an inch in this combined thickness, the external circumference should be reduced by 0·0327 of a foot. It is convenient to make a short table of multiples of this figure, from which may be taken directly the correction for any whole number of sixteenths of thickness. The corrections for 1/32nd and 1/64th of an inch are proportional, and may be given separately, to be added in when necessary.

Later on, when the fully corrected mean internal circumference of a course has been obtained, multiplication of the square of this figure expressed in feet by the factor 0·0413063 gives directly the number of Imperial gallons per inch depth in the course. The result is then used in building up the table over the course concerned.

For new tanks, the plate thicknesses may be taken from the makers' drawings if the tolerances in thickness are low enough not to affect the accuracy required in the tables. The plates may otherwise be measured by suitable calipers before erection, or by weighing, using the known area of the plates and weight per cubic foot of their steel.

If the tank is already erected, the thicknesses given on the drawings may be checked by measuring the exposed edges of the plates. In this it is often necessary to make allowance for the thickening of the edge which results from caulking. Experience helps in this estimate, but if drawings are available, it is better to regard edge measurements as checking these, rather than as measurements to be included in subsequent calculations. Drilling of plates to determine their thickness is seldom practicable.

The plates of tanks which have been in use may show corrosion of varying extent and character. The choice of a good average figure for the thickness of such plates is often a matter of considerable difficulty. If the tank can be emptied, then the extent of any general corrosion can usually be estimated by examining the plates near internal fittings or seam overlaps. Taking the overlaps as example, the inner plate protects the outer plate from corrosion. If the original joint between the two plates is located, the radial distance between this and the general surface of the outer plate near

it is a fair measure of any general corrosion. Several measurements should be made and an average calculated.

If corrosion is of the pitted variety, it is often difficult to make more than an estimate of its effect on the average plate thickness. Magnetic or electrical plate thickness testers might perhaps be used to determine the residual plate thickness, but one usually contents oneself with estimates made with less elaborate equipment.

Paint thicknesses can usually be satisfactorily measured by lifting a few flakes with a penknife from levels over which the tank has been strapped. These flakes are then held against the edge of a finely divided steel rule and their thickness measured. Soft paint, especially if thick, or paint containing many lumps—say, the bodies of insects caught in it when wet—is best cleaned from the tape paths before strapping is begun.

Lagged tanks are best strapped before the lagging is put on. If these come forward for calibration later, it is convenient to calculate their tables from the drawings, or, if this is insufficient, to calibrate them from internal measurements.

Correction for Oil Temperature.

As explained above, circumferences properly measured are those which would have been obtained if the tank and its contents were at the temperature of calibration of the tape. When the temperature at which the tank is to be used differs from this, correction may have to be applied if the tables are to maintain high accuracy.

Heavy fuels are often kept artificially at high and roughly constant temperatures all the year round. For other products the temperature of the oils stored varies with the season of the year, the range of variation depending on the local climate. It is, however, hardly practicable to use more than one table for the same tank. In practice the tables may be corrected to the average temperature of the oil stored. This average may be sufficiently estimated from records at the same installation, or from records at other installations in similar climates.

Correction of measured circumferences for the linear expansion of the tape between its temperature of calibration and the average temperature of the oil would be sufficient for this purpose. In normal gauging, however, the temperature of the dip-tape is also affected. This in turn affects gauges or dips obtained in routine, so that these may reasonably be corrected to the mean tank temperature also.

If the dip-tapes used are standardized to be correct at the same temperature as the tape used in strapping, it is possible and convenient to combine these two corrections and insert them in the tank table. Strappings are then left uncorrected, but the volumes calculated from them are later corrected by means of the cubical coefficient of expansion of steel, using the appropriate temperature difference. The tank table then shows the volumes contained in the tank at its mean temperature of use, and as measured by steel dip-tapes. In the method of calculation preferred by the author, this correction is applied to the volume per unit depth calculated for each course, this being an equivalent and more convenient process.

Correction for Expansion under Oil Head.

Tanks expand under the pressure of the oil contained in them. The effect is negligible in tanks of small diameter, but it increases with the square of the diameter. For large tanks, the increase in capacity on filling with oil, as compared with the volume when empty, may require consideration. If the tank is not normally full on strapping, its total capacity calculated from the strappings will be less than its actual capacity for oil. If, before strapping, the tank has been filled to its normal maximum content, then the resulting expansions are included in the circumferences measured.

Factors affecting the amount of this expansion are the radius or circumference of the tank, the gravity and depth of the oil above the level considered, and the thickness and elasticity of the plate at that level. The usual calculation for boiler expansion may be applied to obtain an exact formula. An approximate formula, which assumes fixed values for some of these factors, but which has met the writer's needs, is given in Bell's "American Petroleum Refining," 2nd edition, page 467. This gives the linear expansion in feet of the circumference as being the product of the square of the circumference in feet and the head of oil in feet, divided by three million times the plate thickness, expressed in sixteenths of an inch. In symbols :

$$E = HC^2/3{,}000{,}000t$$

Since the expansion is proportional to the head H of oil, the average expansion for each course will occur at mid-level if the course is full of oil. Since for any tank of the types considered the circumference C is approximately the same for all courses, $C/3{,}000{,}000$ may be written as K, a constant, when the formula gives :

$$E/C = KH/t$$

E/C is the fractional increase in circumference, and if this is measured or calculated at mid-level in a course full of oil, it is the mean fractional increase in circumference for that course. The mean fractional increase in area of the course is then sufficiently closely $2E/C$, or $2KH/t$. For a course completely filled with oil, therefore, the increase in volume due to oil head will be :

$$v = 2KVH/t$$

where V is the approximate volume of the course, and H is measured upwards from mid-level in the course to the oil surface.

If the tank courses are of equal height, their volumes will be approximately equal. It is often sufficient in this calculation to take the average volume of all courses as the volume for any course, when $2KV$ is a constant for the whole tank, and may be written as R. The formula for any course then becomes

$$v = RH/t$$

For any particular course, the plate thickness t is constant. Taking R/t, which is now constant, as Q, the formula reduces to

$$v_n = Q_n H_n$$

the suffixes being inserted as a reminder that all the quantities will vary from course to course.

Summarizing, the general constant for the tank, $R = CV/1{,}500{,}000$ is first calculated. The course constants Q_n are then calculated by dividing R by the known plate thicknesses for each course. When H_n, which will vary from course to course, is known or assumed, multiplication of Q_n by H_n for all the courses which are completely full of oil, and summation of the resulting values of v_n, gives the total expansion of the tank under the head of oil considered.

The units of volume in which the tank expansion is obtained are of course those in which V has been expressed. The units in which C, H and t are to be taken are given above. The calculation does not consider the expansion of courses which are not completely full, but tables may be satisfactorily corrected without this, as explained below.

It is usual to strap tanks full, and to take this as correcting the tables sufficiently for the expansion of the tank under oil head. When relatively small quantities of oil are delivered from a large tank which is nearly full, however, the removal of the oil reduces the oil head on all courses below. These lower courses contract as a result of this. An error in the quantity calculated as delivered therefore arises when the tank tables have been made on the assumption that the plates of all courses are always fully expanded.

Calculated as a percentage of the quantity delivered, the effect may be substantial, over 0·1 per cent. In the case just mentioned rather more oil is delivered than the usual tables indicate. Since the tables give the full capacity of the tank correctly, it follows that if relatively small quantities of oil are delivered from the lower courses, the amounts delivered are overestimated. Corresponding effects are shown when relatively small quantities are received into large tanks.

By calculating the total expansion of the tank when the oil surface is at the top of the top course, the top of the next course, and so on down the tank, the variations in capacity are obtained at a sufficient number of levels to allow correction of the tables. The way in which this correction is most conveniently incorporated depends on the method adopted for calculation of the tables.

If the tank has been strapped full of oil, all circumferences will have been fully expanded. Course capacities calculated from these effectively contain the corrections applicable when the tank is completely full. Correction at any oil level then involves applying the difference between the correction already contained in the tables, and that properly applicable when the tank is filled only to the level under consideration.

If the tank has been strapped empty, the corrections are simply additive to the capacities calculated without them. It is useful to make the rule that a complete gauge of the oil in the tank is taken at the time of strapping. Tanks may have to be strapped partly full, when the data so given allow adequate correction of the resulting tables. Whether the tank has been full, partly full, or empty at strapping, convenient methods for incorporating the appropriate corrections are not difficult to devise.

Overlaps at horizontal seams strengthen the tank against this expansion. Tanks are less expanded by oil pressure near these seams than above or below them. In those few cases which have been carefully examined by

the author, the effect does not appear to extend more than a few inches above or below the seam. This is what one would expect, since the overlaps are of small depth. The real effect on the tank capacity is very slight, but the fact that it is possible to detect the effect offers another reason for not measuring circumferences too close to horizontal seams.

As a result of the additional resistance to expansion given by the bottom plates and angle iron, Bell's formula is inapplicable near the bottom of the bottom course. Expansion under oil-head is usually not detectable at the bottom of the bottom course, but full expansion in close agreement with the formula is usually obtained towards the top of this course. In calculating the expansion of the bottom course, a reasonable approximation is usually given by assuming that the bottom of the course does not expand, while the top is fully expanded.

Deadwood.

Tanks usually contain " deadwood," or fittings which affect their capacity for oil. Fittings which affect the desired accuracy of the tables must be allowed for, at the levels which they occupy. Swing-pipes are seldom allowed to project from the oil while measurement takes place, so that it is sufficient to allow for these and their incidental fittings at their lowest position. Manholes add to the tank capacity, and must be so treated in the calculations. No attempt need be made here to give a complete list of possibilities : the same principle is applied to all cases. Calculation of the volumes of deadwood, though simple enough, takes time. It is therefore best begun well in advance of the actual strapping. When standard fittings are used, results should be filed for ready reference in subsequent cases.

Internal seam edges project inwards from the inner surface of the plates, and so form a type of deadwood. Internal rivet-heads similarly displace oil. Sufficient check on such small deadwood should be kept, but it will often be found, especially for large tanks, that these small corrections do not affect the required accuracy in the tables.

Handling of Corrections.

For any previously chosen standard of uncertainty in the final tables, it will be found that certain of the deadwood and other corrections given above are so large that their effects must certainly be included. Others will be as certainly negligible, whilst a third class is on the border line between inclusion and exclusion. Care has to be taken with this third class, since, although individually negligible, neglect of their total may affect the accuracy desired.

Tank-Bottom Calibration.

Unless on adequate concrete foundations or iron-work supports, tank bottoms are seldom really flat. Tables are, however, frequently calculated as if the bottoms were flat, even when great irregularity is present; this on the grounds that these lower parts are seldom used in measuring either receipts or deliveries of oil. The stock of oil calculated as being in the

tank will however contain an error owing to this neglect. Where this is important, it may be necessary to correct the tables for the irregularity of the bottom. The method of filling is usually used in such cases.

In the simplest method measured quantities of water are filled into the tank until all bottom irregularities are fully covered, when the resulting depth of water is measured. This gauge or dip and the corresponding measured volume form the first entries in the tank table, no good measurements below this being possible. If at the same time the level of the water relative to the under side of the bottom angle iron, or to the first horizontal seam, is measured, the tank table may be completed to the top of the tank by the method of strapping. The volumes deduced from the strappings are added on to the first volume determined by filling.

A more thorough method is to fill the tank with water till the point on the bottom which will later be touched by the dip-weight in routine dipping is just wet, the volume of water necessary for this being measured. Measured volumes of water are then added, a water gauge or dip being taken after each addition, until bottom irregularities are fully covered. From the tabulated results a suitable table for the irregular part of the tank is readily formed.

Meniscus Correction.

One further correction may be included in forming the final tables. When the oil in a tank is dipped, it touches the steel tape in a meniscus, this reaching about 1/12th inch above the true surface of the oil. Oil depths are therefore always read about 1/12th inch too great. It is however easy to adjust the tables so that the true volume is given against the gauged depth of the oil.

Acknowledgment.

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THE INSTITUTE OF PETROLEUM.

THE One Hundred and Eighty-Fifth Meeting of the Institute was held at the Royal Society of Arts on Tuesday, January 10th, 1939, following the Special General Meeting at 5.30 p.m. The President, Lt.-Col. S. J. M. Auld, O.B.E., M.C., D.Sc., occupied the Chair. Mr. P. Kerr, M.A., B.Sc., A.I.C. (Member), presented the paper on Tank Strapping (pp. 109–124).

MR. P. KERR, in presenting his paper, said that it had been written in response to a request made by Sub-Committee No. 10 (Measurement of Oil in Bulk) of the Institute that he should write out his views on tank calibration, so that they might have a chopping-block, so to speak, on which to work out their own standard methods. He had therefore set out the theory on which his own views were based, rather than the practical methods by which the measurements necessary might be made.

In his own view, the most useful suggestion made in the paper was that of choosing in advance the percentage error, or rather uncertainty, permissible in the final tables. If that was done, it seemed to him that everything else followed. He could not be sure that he had seen everything written on tank calibration, and he made no suggestion that the idea of adopting such a limit was novel, but it was original in the sense that he had thought of it himself.

Some of the uses to which such a limit might be put were as follows. The expected external circumference of any course might be calculated in advance from the drawings, and, in the light of the accuracy required in the tables, upper and lower limits also, within which the actual strappings must lie if they were to be in the required agreement with the drawings. If, on strapping, the circumference found lay within those limits, there was no need to repeat the measurement: it was already checked. Alternatively, if circumference measurements were to be checked by direct repetition, then, even if the duplicate figures differed slightly, further checks were unnecessary, provided that both figures lay within the allowable range. Calculation of the allowable range was simpler in the second application; the check obtained did not depend on the drawings, but a little more work was thrown on the strapping crew. The use of the limit for including significant and rejecting insignificant deadwood or other corrections was obvious. Again, the number of decimal places to which calculations should be taken was that which left the final tables free from arithmetical errors affecting the desired accuracy. In practice, for any particular tank, the accuracy aimed at for its tables might not be fully attained, or it might be slightly exceeded. It was the order of accuracy, rather than too rigid a figure, which he suggested should be chosen in advance.

There was one way in which he thought the limit should not be applied. It was possible to calculate beforehand that measurements to the nearest half inch, say, were all that were necessary to secure the accuracy aimed at, and one might instruct the strappers to read their tape to the nearest

half inch only. To read the tape as accurately as its graduations permitted, however, was really no more troublesome; he felt one should not reject any additional accuracy so easily obtainable. The strapping tape should always be read as accurately as possible, interpolation being made between its graduations. Even if it was found that the circumference figures were a little more accurate and the final tables a little more reliable than was at first intended, surely no one would be disappointed.

There was, of course, a practical limit to the accuracies obtainable. Independent checks suggested that, in normal cases and where strapping was a suitable method, it was easy to make sure of the tables within 1 part in 1000, or 0·1 per cent. Particularly for larger tanks, accuracies of about 1 part in 10,000, or 0·01 per cent., were obtainable with a little more care and attention, but still without serious practical difficulties. That was for the body of the tank; stock figures might be less reliable through uncertainties in bottom calibration; but, as was well known, it was still usually possible to maintain good accuracies in receipts and deliveries. He thought that higher accuracies were outside practical politics. The increase in practical difficulty between accuracies of 0·1 and 0·01 per cent. was not very great, but serious trouble was encountered if one tried to go much further. The list of corrections to which the major part of the paper was devoted was therefore limited to those which, either individually or in total, might affect the resulting tables to the extent of 0·01 per cent. Within that limit, the list was intended to be exhaustive, and he would therefore very much appreciate the advice of the members if he had omitted any correction which should appear in the list.

The importance of any particular correction depended on circumstances, and notably on the local climate. He had tried to consider what might be necessary in any climate, and that was reflected in the paper. In any single case or single climate, the smaller corrections were often inherently negligible; occasionally one or two might cancel out against other corrections of opposite effect.

DISCUSSION ON "TANK STRAPPING."

Mr. J. Kewley said that he had worked in close conjunction with Mr. Kerr for many years, so that Mr. Kerr's views were naturally his views.

He would like, in the first place, to act as spokesman for the numerous people who were going to ask Mr. Kerr questions in thanking him for the excellent paper he had written. The whole subject of the measurement of oil was one of very great importance. Vast quantities were involved. Very often in the past measurement had been done in a slipshod fashion, standard instruments not having been used. When one looked into the subject, one was surprised at the large number of pitfalls that existed. For example, a tank of oil when measured empty had a smaller capacity than when measured full, because of its expansion under the heavy liquid. There were many points like that which were of importance.

He thought that the subject of the paper, which had arisen in connection with the work of Committee 10, of which Mr. Kerr was an active member, was one which should be followed up and one which gave the Institute an opportunity of taking a definite lead, because, as far as he knew, very little had been published on the question of tank measurement and measurement of oil generally. He hoped that eventually Committee 10 and the American Petroleum Institute, for example, might come together and try to arrive at some general agreement on methods of measurement. So far success had not been achieved by the attempts to obtain international agreement even on testing methods.

With reference to the statement in the second paragraph of the paper, "It is difficult to measure the effect of corrosion in the plates of old tanks without this," *i.e.*, without emptying the tank completely, he had seen at the Physical Exhibition two years ago a small apparatus with which the thickness of a plate of iron could be measured by means of changes in magnetic flux, and he thought that possibly one could explore the outside surface of a tank in that way and get some idea of the extent to which corrosion had been taking place.

Mr. Kerr said there were electrical instruments which enabled the thickness of a tank plate to be determined, but, so far as he had looked into them, they scarcely seemed to offer the accuracy that was desired. The line along which he thought internal corrosion could be most easily tackled—a line which he had not yet had time to explore fully—was as follows. The expansion of a tank under oil head depended on the thickness of the plate; if the lower courses of a tank were strapped with the tank empty and also with the tank full, inversion of the usual boiler expansion formula enabled one to calculate the actual plate thickness.

Mr. H. Hyams, referring to the statement made by Mr. Kerr in his remarks that evening, that with his method of tank strapping he could obtain an accuracy of 1 part in 10,000 in the case of large tanks, said it would be interesting to know what evidence Mr. Kerr could bring forward in support of that claim.

Mr. Kerr said it was difficult to obtain independent methods of check which would verify accuracies of the order in question. The first trials that he had made were to determine how closely he could repeat the measurements of the circumference of a tank. At that time he had a tape which was only 50 ft. in length, and he strapped a tank the circumference of which was a little less than 250 ft. in five sections. He set out five fine vertical lines around the tank circumference and measured the intervals between these, adding the measurements together to get the total circumference of the tank. Taking a new set of lines, he repeated the process; the manager of the installation, who was interested, repeated the measurement twice independently, again with new zero lines. On the total circumference the greatest difference between any pair of the four results which the manager and he obtained was only 1/32 in. That was perhaps accidental, as they were working under very good conditions, strapping a level which was easily accessible and not a less accessible level higher up the tank, but it at least showed that the fundamental part of the measurement, the circumference, could be taken to a high degree of accuracy.

Again, he had strapped tanks in France, where the original tables were, of course, made by the French Bureau of Weights and Measures, and he had found that his results usually came within 0·01 or 0·02 per cent. of the figures which the Bureau had obtained. That might be challenged, because the Bureau also calibrated the tanks by measurement; it might be held that this proved merely that methods of strapping were self-consistent.

At one installation which he had visited, fuel oil deliveries were weighed in rail cars, and as compared with the amounts recorded as being taken out of the storage tank, the installation was gaining on its deliveries, the gain shown being 0·35 per cent. On re-strapping the tank he found that the tables under-estimated its capacity by 0·33 per cent. Again that agreement was perhaps a little accidental, because, in order to obtain it, he had to see that the weighbridge used by the installation was in nearly perfect order and, of course, there were always difficulties in weighing rail wagons; the wind had an effect on their weights, and if the weather was wet that had an effect also. He thought, however, that the agreements which he had just set out indicated that the order of accuracy obtainable on large tanks by strapping was about 0·01 per cent.

Dr. F. H. Garner asked Mr. Kerr what apparent change in volume was obtained if a measured volume of oil in a tank calibrated by his methods was transferred to another calibrated tank?

Mr. Kerr said he had not carried out many very close transfers of oil. The question of errors in gauges and errors in temperature was so troublesome that measurements of that sort would have to be repeated several times with the best attainable pre-

cision before a check was obtained on a method which seemed to give an accuracy of about 0·01 per cent. Fuel oil would have to be used, so that there would be no loss by evaporation; otherwise there would be a difficult correction to be made for the amount of vapour lost in transfer. He had no sufficiently accurate set of figures for such transfers; they did not promise enough in the way of checking.

Mr. W. F. Jelfs said he believed it was not the general custom to apply corrections for expansion under oil-head, and he would like to know whether Mr. Kerr considered that that should be the practice in future.

Mr. Kerr said it would be seen that the paper did not suggest any particular standard of accuracy which should be adopted in the calibration of tankage. The need for the correction of tank tables for the expansion of the tank under oil-head depended on the standard of accuracy that one wished to attain in the tables themselves. It was only for larger tanks that this correction was of much importance, but, as he had explained in the paper, it was of rather more importance than it appeared to be at a first glance, since when a small quantity of oil was delivered from the upper levels of a large tank, contraction of the plates took place on all courses below the level from which delivery had been made. As a result of neglecting the expansion correction, there might then be an unexpectedly serious percentage error in the quantity calculated as delivered or received. He would like to see as good accuracy as was commercially possible in oil measurement, and therefore he favoured the insertion of an expansion correction in all cases where that seemed to be justified.

Dr. A. I. V. Underwood asked whether in the formula given in the paper for expansion under oil-head—*i.e.*, $E = HC^2/3{,}000{,}000t$—it was the depth of oil that was referred to or the pressure head of the oil. The latter would vary with the specific gravity of the oil; the correction required would be different, according to whether the tank was used for petrol or for heavy fuel oil. The effect of the correction for expansion under oil-head was given by Mr. Kerr as being approximately 0·1 per cent. in certain circumstances. If one took the difference in the specific gravity of petrol and heavy fuel oil—a difference of roughly 25 per cent.—the error due to that difference in specific gravity would be 1 in 4000. That would be appreciably larger than the error of 1 in 10,000 which Mr. Kerr regarded as the ideal.

Mr. Kerr said that the statement in the paper: "Calculated as a percentage of the quantity delivered, the effect may be substantial, over 0·1 per cent.," applied to relatively small deliveries from the tank. The total correction, considered as a percentage of the tank contents, reached 0·05 or 0·07 per cent. on large tanks only, so that the variation was a little less than Dr. Underwood had just suggested.

He was glad that attention had been directed to the point in question, because he had already been told that he ought to apologize for inflicting elementary mathematics at such great length on so distinguished a body as the Institute of Petroleum. The discussion on the correction for expansion under oil-head was very simple indeed, and it could have been condensed, but he had a certain reason for setting it out at some length. It was clear that it was not every tank that could be treated in the way described. Some tanks had courses which were not equal in height, and the user of the formula might depart from the calculations given in the paper at any particular stage of the discussion. He had therefore put in each stage in full, so that anyone who wished to calculate the expansion more accurately than the approximate formula allowed could easily see the point at which he must depart from the procedure suggested in the paper.

He accepted Dr. Underwood's comment in principle. It was true that the formula in the paper was approximate only; it was derived from the exact formula by taking 0·750 as a fixed gravity for the oil, a mean value being also taken for Young's Modulus. The formula given did not balance in dimensions; the constant 3,000,000 necessarily contained factors which had dimensions.

As suggested by Dr. Underwood the accuracy of the formula given would be improved by taking in its place

$$E = HC^2g/2{,}250{,}000t$$

where the new factor g is the specific gravity of the oil, the other quantities being as given in the paper.

The general formula is

$$E = pC^2/2\pi tM$$

where E is the expansion of the circumference, p the pressure due to oil-head, t the plate thickness, and M Young's Modulus for the steel plates, at the level concerned. Either of these may obviously be applied as suggested in the paper.

Dr. M. B. Blackler congratulated Mr. Kerr on ventilating a subject which was of undoubted importance to the oil industry in general. He did not believe that questions of head, expansions and contractions of tank materials seriously affected commercial measurements. Tanks as manufactured to-day, when transported and erected under reasonable conditions, could be calibrated within the limits of commercial accuracy from the makers' drawings, but if erected abroad after the plates had been transported long distances by sea, rail and road, then calibration by direct measurement was essential. In such cases he had adopted the following plan in order to obtain reliable calibration figures. First levels were taken over the bottom of the tank, and the general configuration was drawn out so that the calibration of the first foot of height could be determined with reasonable accuracy. The diameter of each strake of the tank was measured at the top, middle and bottom, and these diameters were measured at angles of 45° round the tank, so that actually twelve diameters were measured for each strake and the average diameter was taken as the accepted diameter of the strake. The calculations were made in litres per centimetre, and as a matter of routine, allowances were made for volumes of rivet heads, angles, ladders, etc., in the calibration volumes per centimetre.

The work was included in the drawing-office routine, and when completed the calibration tables were distributed from that Department.

Dr. Blackler directed attention to calibration difficulties in those cases where the tank bottom moved under varying oil-head, mentioning a case in his experience in which the ground under a tank was found to be saturated with oil which had leaked through the tank bottom as a result of this movement.

Mr. Kerr said he quite agreed that the calibration of a tank bottom was difficult if the foundations were not sound. Movement did occur in particular cases, as in that mentioned by Dr. Blackler, where it had led to leakage. The suggestion that movement was taking place was, however, sometimes made without very clear proof, often as a rather speculative explanation of discrepancies in measurement; in these cases one should estimate probabilities from the nature of the ground and the foundations. In doubtful cases it was well to make sure, for more than measurement was involved; as Dr. Blackler's remarks implied, the tank should then be watched for possible leakage. To meet both leakage and measurement, he suggested putting a temporary water bottom in the tank, sufficient to cover the bottom completely, and determining by dipping whether the level of the water surface changed with the oil-head.

This could be done without taking the tank out of use, and without much trouble, since routine water dips taken from day to day would usually give the information required. If the tank bottom had already begun to leak, the expected relation between the water dips and the oil-head might be complicated by actual loss of water, but by prolonging the trials until the tank had been filled and emptied twice or thrice, the point would be cleared up. If movement were confirmed, the water bottom might be left in the tank, when gradual disappearance of the water would show that leakage had begun; measurement errors would be reduced also. If the water put in was sufficient to keep its surface above the bottom plates even when the tank was full of oil, then it would be possible to calculate from the water dips the volume added to the tank through depression of the bottom, and so estimate the seriousness of the movement taking place; the figures obtained should be regarded as a minimum, since the point at which the water-finder rested on the tank bottom might itself move downwards with increasing head of oil; but this movement could in turn be measured and allowed for by recording the varying heights from the edge of the dip-hole to the point in question.

As regards Dr. Blacker's method of tank calibration, he agreed that tanks might be satisfactorily calibrated in various ways. Calibration of tank bottoms by survey methods led easily to a figure for the total content of the tank bottom to some chosen

depth of liquid; from such data it was possible, but perhaps less easy, to form a table allowing measurement in the tank bottom itself of small quantities of water, say those accidentally delivered with a cargo. The methods of filling outlined in his paper were useful when bottom calibration had been omitted when the tank was first erected, but was later found desirable; for it was generally possible to pump in measured quantities of water under the oil, and to measure the resulting depths with water-finding paper or paste, without taking the tank out of use.

That the expansion of tanks under oil-head could always be neglected was, of course, the current opinion. His own view was that the need for this correction varied with circumstances; it was only necessary with large tanks. When even a large tank was gauged only when it was nearly full or nearly empty, as might be the case with tanks used only for cargo shipments, then the effect was often sufficiently allowed for by strapping the tank when it was full, without further adjustment of the tables. When small deliveries were made out of large tanks, the effect was more serious, as the paper suggested, and as could readily be calculated. His own view was that in these cases at least, a correction so readily calculated and so easily incorporated once for all in the tank table should scarcely be omitted.

Mr. A. P. Catherall said he would like to ask Mr. Kerr what his experience was with regard to the compensation of the various errors that entered into the measurement of oil. The calibration of a tank was only a part of the measurement of the oil; there were irregularities in tank dippings, temperature measurements, and so on. His own experience was that there was a number of such errors which generally cancelled themselves out, so that, even with quite casual tank strapping, and even in taking the measurements from the blue print, provided that they were checked with at least one strapping, one could obtain quite accurate results without extremely accurate tank strapping.

Mr. Kerr agreed with Mr. Catherall in thinking that strapping was often unnecessary if the tank was well made and the plates were undamaged. Official regulations, however, often required the tables to be made from direct measurements, so that one was compelled to strap more frequently than was necessary on purely technical grounds. Calculations from drawings were usefully checked by strapping a single circumference, as Mr. Catherall had suggested; that this was his view would be seen in the paper.

In strapping, the length of tape required to encircle the tank was a minimum when the tape followed its proper path; it followed that small departures of the tape from its proper path could only have second-order effects on the tank tables, and this was thought to be a reason why Mr. Catherall had obtained good results without extreme care. It also followed that if care had been taken with the tape tension, there was something to be said for taking the smallest figure obtained for any given circumference, rather than the average of repeat figures, in calculating the table; but if tolerances had been calculated as suggested in the paper, and all repeats fell within these, their average, or indeed any of the figures, could be taken into calculation without affecting the accuracy desired.

When discrepancies in measurement were troublesome, it was sometimes necessary to find out how much of these should be attributed to errors in the tank tables; here the abbreviated method suggested in the paper under "Recalibration" was often sufficient.

To discuss the question of compensation of errors in oil measurement fully would lead too far from the subject of the paper, but Mr. Catherall's experience on this question was also his own, and it was in line with theory. He had found that errors in oil measurement followed the usual mathematical theory of errors; given a sufficient number of cases, it was usually possible to separate the effect of systematic errors from that of fortuitous errors. The latter were usually distributed normally; in the normal distribution small errors were much more frequent than large, in line with Mr. Catherall's experience. His view was that systematic errors should be eliminated from oil measurement as carefully as possible; much larger fortuitous errors could be tolerated, since they occurred only occasionally, and since they tended to cancel out, as mentioned in the paper under "Preliminaries." Errors in tank tables tended to be systematic in their effect.

Mr. H. Hyams said that Mr. Kerr indicated in his paper the correction to be applied for the difference between the calibration temperature of the tape and the average temperature of the oil that was going to be stored in the tank. He quite agreed that such a correction was necessary and should be incorporated in the tank tables, but he thought it might be borne in mind, in connection with measurement on a weight basis, that hydrometers in the oil industry were usually calibrated at 60° F. or 15° C., and they were frequently used in tropical climates or in connection with artificially heated oils without any correction at all being made for the cubical expansion of the glass. The error which would result from neglecting that correction was, in the case of an oil whose specific gravity was determined at, say, 90° F. about equal to the combined corrections that Mr. Kerr said were necessary under the heads of expansion of the tank and also expansion of the tape. He saw no way of meeting that contingency other than that the hydrometer should itself be calibrated at a temperature corresponding to the oil with which it was to be used, or, if the standard hydrometer calibrated at 60° F. was used, it seemed to him that, in those cases in which the oil was at a temperature substantially above 60° F., the appropriate correction for cubical expansion of the glass should be made; otherwise the correction which Mr. Kerr indicated was necessary would be nullified.

The importance of correct calibration of the tank bottom, where irregularities are known to exist, has received due emphasis in the paper. The provision of a permanent water bottom is a remedy, but there are many countries where very cold winter conditions make such a water bottom undesirable or where water bottoms are, in any case, prohibited by local authorities. In such cases the correct measurement of oil quantities in tanks with deformed or weak tank bottoms becomes a real difficulty, and this difficulty can be overcome only by careful calibration. The shape of the bottom and its cubical contents will possibly vary with the head of oil; it has been suggested that with certain types of tanks the bottom acts as a diaphragm, although conclusive evidence has not been forthcoming to support this contention. But in such instances it would be undesirable to construct tables for the bottom levels from the tank drawings. Mr. Kerr's method of bottom calibration by water could be followed when the new tank is submitted to its water test.

There was one further question that he would like to ask. Mr. Kerr said that it was desirable to strap tanks when the temperature of the air and the temperature of the contents of the tank were approximately the same. It could not be simple to obtain a predetermined air temperature, and he would like to know how that difficulty was overcome by Mr. Kerr.

Mr. Kerr said that Mr. Hyams had raised an important question in his first point, whether measurement should be considered as a whole or sectioned off into questions of tank calibration, dipping, taking specific gravities, and so on. He thought that tank calibration could perhaps be more fairly relegated to its own department than the rest of the routine process of gauging. It was true that one might offset the effect of the neglect of the expansion of glass correction for the hydrometer against tank-table corrections, but Mr. Hyams had himself suggested a better method. If tanks were correctly calibrated, one should not rely on compensation of errors, but should rather correct the hydrometer for its errors when used at a high temperature or, alternatively, for high temperatures supply a hydrometer which required no correction. Generally speaking, he was opposed to allowing errors to compensate each other, unless one was quite certain that compensation would be reasonably perfect. Again, although in some countries measurement was by weight, in others it was by volume, when the compensation proposed would fail. It was possible to have one method of tank calibration for areas where measurement was by weight and another method for areas where measurement was by volume, but it was rather doubtful whether it was advisable to duplicate methods in that way for the sake of compensation of errors.

The last point raised by Mr. Hyams was a very difficult one, and one on which he would admit that he was not himself quite satisfied. It was very difficult to arrange that the air, oil, and tape temperatures should all be the same. The linear expansion of steel was about 0·000012 per degree C., so that if the tape temperature was 8° C. away from the temperature of calibration of the tape—the temperature at which he assumed the tape was correct,—an error would be introduced of about 1 part in 10,000

in the circumference measured. An error of that amount in the circumference corresponded to an error of 2 parts in 10,000 on the cross-sectional area; in order to maintain an accuracy of 1 part in 10,000 in the tank tables, one would have to be sure that the tape was within 4° C. of its correct temperature, or else measure in some way the temperature at which the tape was being used. Neither of those was very easy. The simplest course to adopt, of course, was to reduce the accuracy of the tables, to choose such a tolerance as would cover the errors that were made with the tape. An alternative method was to substitute a tape of low coefficient of expansion for the normal steel tape used. It was true that in the paper he had expressed himself as being opposed to the use of such special tapes, but in the case in question one was confronted by a special difficulty and had to choose between two undesirable courses.

The tape temperature was important in the tropics. There, if in full sunshine a tape was put round a tank which was painted or had a pitted surface or was rusty, when one drew one's finger along the tape the sensation was not merely one of warmth, but of slight pain. The tapes might reach temperatures of 140° F. or more under these conditions, which was quite a departure from the 68° F. at which they were normally calibrated. He hoped that with further work some method might be found of correcting for the major portion of the error introduced by failure to control the temperature of the tape. As an illustration of what might be done, one might be able to hang a thermometer of some standard type on the sunny side of the tank and, by strapping a few tanks both with an ordinary steel tape and with an invar tape, obtain a satisfactory table of corrections, to be applied in future cases against the readings of a similar thermometer similarly placed during strapping.

Except for tanks heated artificially, in all climates there was always a time in the morning, with another in the evening, at which the air, oil and tape temperatures would be the same; but it was extremely inconvenient, and often impracticable, to have to limit strapping to these times. He agreed that the point was a difficult one, not yet fully solved.

He agreed that insertion of permanent water bottoms would be impracticable in very cold climates; in such cases check on the stability of the tank bottom might still be possible in summer. The fact that movement of the bottom was apt to start leakage, combined with the impossibility of maintaining all the year round a water bottom against leakage, would no doubt lead engineers to make very sure of tank foundations under such circumstances. While he had not had to handle the specific case of inserting a temporary water bottom when local regulations did not allow these, he had seldom found any real difficulty in obtaining temporary relaxation of official regulations, of course within reason, after the need for and intention of the work proposed had been suitably explained.

Mr. W. E. Douglas said one point to which Mr. Kerr had not referred in this paper was that there were limitations with regard to tank-calibration tables. There were certain persons who were responsible for those limitations. In the first place, the tank-calibration table was limited according to the limitations of the person or persons making the measurements. It was unfortunate that the people who had to calibrate tanks had not always the fine, scientific, accurate mind of Mr. Kerr, and it was very difficult to get them to realize the importance of some of the finer points. One might possibly produce more accurate results by not aiming at such a high degree of accuracy. But the limitation which still more affected the tables was the limitation of the people using them. If the tables were too complicated or required too much judgement on the part of the people using them, it would invariably be found that the tables were wrongly applied. One of the greatest difficulties in attaining a high degree of accuracy was the fact that the mental calibre of some of the people using them was not sufficiently good, and that, of course, was particularly the case in places far overseas. He therefore thought it was advisable not to aim at too high a degree of accuracy if that involved making the tables themselves too complicated.

Mr. Kerr said that if one had not at one's disposal staff who were competent to strap a tank properly, then the results would be unreliable. To send out a special crew to remote districts might be out of the question, on account of the cost. In practice one might have to admit that in such cases one could not reach satisfactory

accuracies by strapping. In these cases calculation from the drawings would almost certainly be more satisfactory than strapping.

He was entirely in agreement with Mr. Douglas as regards tank tables. These state against the dips the calculated volume of oil in the tank; in his opinion, the volume figures given should already contain all corrections considered desirable. For the type of tank considered in the paper, any desired correction could be incorporated in calculation into the volumes given, this leaving one completely free to set out the table in any desired form; he agreed with Mr. Douglas that the user should be given as simple a table as possible.

MR. J. LEWIS said that Mr. Kerr stated in his paper that the strapping tape should not come within four inches of a seam. He would like to ask Mr. Kerr how he arranged for that, because in his own experience of strapping it had been difficult enough to keep the tape on top of the plate in its correct position.

MR. KERR said that although he had not met the trouble, it might be difficult to maintain a tape in correct position on a tank, but if the circumference was to be of value, it had to be done. The tape was put round the tank in tension, and it was moved into place by means of a soft metal ring which passed round the tape. A string was led from the soft metal ring to the top of the tank and another string was led to the ground. Two men at the ends of the string were able to place the tape in a satisfactory position. Ten pounds tension on the tape was generally enough to hold it in position, but if this difficulty was experienced with any particular tape, he would increase the tension until he got the tape to keep in place. A correction for the additional tension might then have to be applied, but since the same tape would probably have to be used on other tanks, this would be calculated as a percentage correction once for all.

Using a tape ¾ in. wide he had not met serious difficulty in keeping it to the proper path, even when the two men assisting him were natives new to the work. The wider the tape the more easily it would keep to its path, once it had been placed in this; the solution might be found in using a wider tape, but on this see the section on "Strapping Tapes" in the paper.

MR. J. LEWIS said his experience was that as soon as one increased the tension, the man at the other end of the tape invariably let it go. He attempted to keep the tape some distance above the horizontal seam, but he found that it came down on the horizontal seam, and he had to go round the tanks himself to see that the tape was actually in position. He gave up all hope of ever finding the tape 4 in. above the seam.

MR. KERR said that in the method which he preferred the tape was long enough to encircle the tank completely. One end of the tape was held through a spring balance at the desired tension, and the other end was just held in the hand. The friction necessary to pull the tape out of position was about 18 lb., on a tank of fair size, so that at 10 lb. one still had a margin between the amount necessary to pull the tape out of position and that which gave the required tension. He had not found the difficulty in actual strapping, but it no doubt depended to a considerable extent on the quality of the assistance that one had.

A strapping crew must, of course, include at least one man who understood thoroughly what was to be done; it was useful if the second man also understood, since this saved time; but he had frequently had to train as second hand a reasonably but not unusually intelligent native leading-hand or foreman new to the work, this adding perhaps half an hour to the time taken. The third man need have very few qualifications beyond being willing to do as he is told; but this is essential.

The use of a long tape puts both ends of this in the hands of one man, who should be the best qualified of the three. The tape is kept at the required tension by the leader while it is being finally placed by the two others. If it is desirable that the leader should inspect the placing of the tape, and if the third hand is not to be trusted to keep it at proper tension, the following procedure has been found useful. The ends of the tape already overlap; still under the proper tension, the overlap is extended, so that a fair length of the one end lies closely above the other on the tank surface.

The third man is not allowed to take the tape in his hands, but holds it in position by pressing his hands flat against the tank over the two ends. Marks have already been made against graduations on both ends of the tape, and it is pointed out to him that the tape must still be at these marks when the leader comes back. The position of the tape is then inspected by the leader, any faults being pointed out to the second man. The leader again takes over the tape from the third man; before the third man removes his hands, the proper tension is restored on the free end through the spring balance. The tape is then readjusted if necessary by the second and third hands, and its placing again verified; if the process is insisted on carefully at the beginning, it is seldom necessary to carry it out on all courses. If the tolerances within which the measured circumferences must lie in order to agree satisfactorily with the drawings have been calculated in advance, then as the leader comes to each new circumference he knows at once whether further inspection of the tape path is desirable.

Mr. A. P. Catherall said he had strapped a considerable number of tanks with 500-ft. tape, ¾ in. wide, and using coloured labour, he had had no difficulty at all in keeping the tape on the tank at the required tension by the method suggested by Mr. Kerr.

Mr. Kerr said his difficulty generally has been to prevent the men pulling too heavily on the ends of the tape. He had seen them pulling at the ends of the tape as though they were engaged in a tug-of-war, and that had, of course, to be stopped. As he had said, a great deal depended on the quality of the assistance available.

He thought it might be useful to explain more fully the troubles which arose from accepting too low a standard of accuracy in tank tables. Any faults in tables which tended to short-delivery to customers would not be tolerable to any reputable Company. Faults in strapping delivery tanks could be divided on this basis; slackness in the tape, neglect of seam overlap corrections, and the like, tended to short-delivery; excess tension in the tape, if not corrected for, tended in the other direction; while there were other faults whose effect varied according to circumstances. Another consideration was this, that it certainly paid to control working losses closely, at any rate when the quantities involved were large. If a tolerance of say plus or minus 0·1 per cent. were accepted, when a little more care would give higher accuracy, the resulting confusion in working losses was often serious. Two tanks at the same installation might then differ by 0·2 per cent. in the extreme case; if these tanks received oil from tankers, then the apparent transit losses to the installation could vary by 0·2 per cent., even if the real losses were constant in percentage. The performance of the tankers would come under suspicion, and one might easily waste time in looking into this, when the fault really lay elsewhere.

Drawing oil from one tank instead of the other, the installation would show 0·2 per cent. difference in working loss. Local staff would become accustomed to apparently inexplicable variations; if they did not appreciate the part their tank tables were playing, their efforts to produce comprehensible loss figures would be ineffective, when interest in the question tended to be lost. The extreme ranges in working losses often reported from two installations working under closely the same conditions would be familiar to his audience; poor tank tables were often a factor in this.

Although he himself regarded the actual process of strapping as one requiring care and little else, he pointed out that it seemed to be worth while overcoming whatever difficulties might be met with, considering that the resulting tables might be in use for many years. Tanks seldom lay idle; taken over a few years, there were few cases in which the oil volumes dealt with by a table were not large. He would not attempt to lay down what standards of accuracy should be adopted; accuracy for its own sake was a mistake, but in strapping tanks it seemed to him that it promised definite economies.

Mr. B. C. Ferguson asked whether Mr. Kerr had any experience of the correlation between calibration by internal diameters and by external circumferences.

Mr. Kerr said that the method of strapping met his own needs so much more closely than the method of measuring internal diameters that he had not had much

experience of the latter method. He saw no reason, however, why the measurement of internal diameters should not give one an excellent tank-table. It was clear that if a tank was measured empty the diameters obtained did not contain the correction for tank expansion which he preferred should be added in the case of large tanks, but that could be calculated and allowed for. He believed that very close agreement would be obtained between the two methods.

Mr. Malcolm Browne said there was one question he would like to ask with regard to buried tanks. Tanks above ground were usually circular, vertical or horizontal, but in the case of buried storage other shapes were used. Had Mr. Kerr had any experience of calibrating tanks that were square or rectangular, for instance, and, if so, what difficulties might be expected in connection with them?

Mr. Kerr, in a written reply to the above question, said that he had had only a little experience with rectangular tanks. Calibration methods that suggested themselves were calculations from the drawings, measurement of internal dimensions, or direct filling with measured quantities of liquid, since rectangular tanks were usually small. He thought that direct filling was likely to be the most accurate method; especially if their dimensions were large, flat ends and sides tend to distort rather notably outwards under the oil head, unless they are very well supported by the earth outside the tank, or otherwise strengthened. He believed he had seen in the literature a discussion of the amount of this distortion, but at the moment of writing he was abroad, so that he could not give a more definite reference; he had made no trials to compare the results of the various methods of calibration on this type of tank.

If the tank were laid with a tilt for drainage, or if it developed a tilt through weakness in the foundations, this might lead to inaccuracies unless allowed for; with small tanks, errors of a few gallons were high in their percentage effects. For this reason, deadwood had to be treated carefully if good percentage accuracies were to be obtained from measurement methods in these cases. Once the tank had settled on its foundations, calibration by filling would allow automatically for tilt effects.

In measuring internally, the end loop of the tape might be held against one side of the tank, and a graduated steel rule in the required position against the other side; the tape would then be read off opposite some convenient graduation on the rule, the tape and rule lengths being added to obtain the total length. The tape might be used similarly between two rules. It should not sag between the tank ends; it might be supported on a wood batten. Small variations from the correct tape tension would not be important for short lengths; but when large diameters were measured, special arrangements would have to be used to secure the proper tension and to permit correction for sag, if good accuracies were required.

Mr. A. C. Hartley asked whether Mr. Kerr had had any experience with welded tanks, and whether there was any difference in the behaviour of welded tanks and riveted tanks in regard to expansion under a head of oil.

Mr. Kerr said he had had very little experience with welded tanks, but he was credibly informed that the welded tank was often less regular in shape than the riveted tank. The only cause of difference in expansion under oil-head, he thought, was that at the horizontal seams of a riveted tank the plates overlapped and at that level there was a greater resistance to expansion than in the open plate. In the case of welded tanks there were no such overlaps, so that he would expect the expansion of a welded tank to follow the boiler expansion formula more closely than would a riveted tank, unless it had stiffening rings. He had yet to calculate out the effect of the overlap of horizontal seams on the expansion; the theory underlying the calculation was rather complex, and he had not yet had time to go into the question properly. He had checked up the effect practically; his results suggested that at four to six inches above the seam the expansion formula given in the paper could be applied satisfactorily. At the seam itself the restraining effect of the overlap could be detected, but it was much smaller than would be expected if the overlap extended over any great depth. It was for that reason that he suggested strapping 4 to 6 inches above the bottom of the course.

DR. F. H. GARNER said that in connection with correction for oil temperature Mr. Kerr stated that the volumes calculated from the strappings were later corrected for the cubical coefficient of expansion of steel. Suitable corrections could quite well be incorporated in the standard volume correction tables; adoption of this procedure would considerably simplify accurate calculation of volumes from tank measurements.

MR. KERR said that Dr. Garner had referred to another side of a question already raised in the discussion—*i.e.*, as to how far oil measurement should be considered as a whole and how far it should be separated out into its constituent parts. It was quite possible to allow for expansion by adjusting the coefficient used for the oil; it was largely a matter of opinion, but, on the whole, he would like his tank tables to show true volumes directly. He was quite willing to agree, however, that the final results would be the same.

Dr. Garner's suggestion assumed that oil volumes would be reduced in routine by suitable tables to 60° F., or at least to some standard temperature. While this was standard practice in the United States system, measurement by weight, without reduction of either volumes or gravities to standard temperature, was common in both the British and metric systems. Here the observed specific gravity of the oil was corrected to the temperature of the oil in the tank, and the oil volume, measured at the same temperature, was multiplied by the figure so obtained to get the weight. The process had the advantage that the effect of possible errors in the coefficients used to reduce the gravities were greatly minimised. Allowance for thermal expansion of the tank in the manner suggested by Dr. Garner was inapplicable when this method was used.

Apart from this, the method of correction suggested by Dr. Garner would be advantageous, since instead of adjusting the tank tables to an average temperature of use the correct allowance would be made at each gauge for the tank's actual temperature. He apologized for having suggested at the meeting that the two methods were equivalent; they would average out equally, but Dr. Garner's method undoubtedly had the advantage in the individual cases.

THE PRESIDENT, in proposing a hearty vote of thanks to Mr. Kerr, said he was sure everyone present would agree with him that the discussion that evening had been most enlightening and interesting. He thought the method which Mr. Kerr had adopted of answering each question as it was asked was a useful one, and might be adopted in many other cases; it had certainly brought out a very large number of points which might not otherwise have been raised. He would remind the members that remarks in writing would also be welcomed, and he was sure that Mr. Kerr would be pleased to answer in writing any questions which were asked in that way.

He thought the Institute was to be congratulated on being able to give the lead to which Mr. Kewley had referred by having a paper on the subject of tank-strapping in its *Journal*. The subject was not an aspect of petroleum technology to which he himself had devoted much attention; he had always been content to leave it in other people's hands, and he had been surprised that evening to learn the accuracy with which measurement of oil in tanks could be carried out.

He wished to express the thanks of the Institute to the Asiatic Petroleum Company for permitting the publication of Mr. Kerr's work, and thus helping the Institute to give a lead on the subject of international standardization of such work. He felt that the prestige of the Institute would be greatly enhanced by the publication of Mr. Kerr's paper and the tables which should accompany it. If any arrangement could be made for coming to some agreement with the American Petroleum Institute on the question of the standardization of measurement, it would be one of the finest ways of creating reciprocal understanding between the petroleum technologists of the world. The Institute was much indebted to Mr. Kerr and his Company for the presentation of the paper that evening.

The vote of thanks was accorded with acclamation, and the meeting then terminated.

ENGINE KNOCK AND ITS EFFECT ON FUEL DEVELOPMENT.*

By Professor D. M. Newitt, M.C., D.Sc., D.I.C., A.R.C.S.

It is my purpose this evening to give some account of the combustion of hydrocarbons and, more particularly, of that aspect of their combustion which is peculiar to the internal combustion engine, and which, in certain circumstances, gives rise to the phenomenon of knock or pinking. The subject is one of considerable technical importance, for the incidence of knock in petrol fuels is a dominating factor in the design of engines and has exerted a profound influence on the trend of development in the petroleum industry.

The origin and cause of knock has long puzzled both chemists and engineers, and attempts to find an explanation which will account for all the facts have hitherto not been entirely satisfactory. All the evidence, however, points to it being a chemical phenomenon resulting in a sudden acceleration of the rate of combustion in the engine and the initiation of intense compression waves in the charge. A knowledge of the chemical changes occurring during a working cycle is therefore an essential preliminary to an understanding of the problem, and I propose to give a brief account of some recent researches which have a direct bearing upon the matter.

It is well known [1] that a quiescent combustible gas–air or oxygen mixture of suitable composition will, under certain conditions (*e.g.*, when ignited in a horizontal tube closed at one end), propagate flame at a uniform slow rate of the order of 5 to 10 metres per second. Provided the tube is sufficiently long the uniform propagation may be disturbed by vibrations which cause compression waves to traverse the medium, and it may eventually develop into detonation, the flame then travelling at the enormously enhanced speed of some 3000 metres per second. These are what may be termed the normal types of flame propagation, and they may conveniently be studied by photographing the flame on a film moving at right angles to the axis of the tube.

There is no need to enlarge upon these types of combustion, which have frequently been described and are probably familiar to you all, but I should like to draw attention to three photographs taken by my colleague, Mr. R. P. Fraser, which illustrate certain points of interest in connection with them.[2]

Plate I (*a*) is a photograph of flame traversing a carbonic oxide–oxygen medium in a horizontal tube closed at both ends. It shows very clearly the way in which a series of compression waves may be set up and traverse the gases.

Plate I (*b*) illustrates the setting up of detonation at two points each a little ahead of the flame front in a similar mixture burning in a horizontal

* Paper presented to a Joint Meeting of the South Wales Branch of the Institute of Petroleum and the local section of the Institute of Chemistry, held at Swansea on December 9th, 1938.

tube. It is interesting to note that from the point of detonation two flames travel forwards, the one at a comparatively slow speed of 360 metres per second and the other at about 3000 metres per second.

Plate II (*a*) shows a detonation wave traversing a carbonic oxide–oxygen medium at a uniform speed of 1760 metres per second. The regularly distributed striations in the photograph indicate that the detonation head is moving in a spiral path, and measurement shows that it is rotating at a speed of 44,000 revolutions per second.

Now the type of combustion producing knock, although associated with compression waves and a high rate of reaction, cannot be identified with either uniform slow propagation or with true detonation. This is shown by Plate II (*b*), which is a photograph of the flame traversing an engine cylinder in which knock is taking place.[3] It will be seen that towards the end of the stroke, when only a relatively small proportion of the charge remains unburnt, there is a sudden increase in velocity accompanied by a violent vibratory movement of the gases. This vibratory movement, together with resonance effects in the cylinder walls, gives rise to the familiar sound of knock.

Let us consider for a moment the condition of the unburnt charge near the end of the stroke. It is in contact with the hot walls of the combustion chamber and with the faces of the exhaust valve, it is subjected to adiabatic compression, and it is irradiated from the flame and bombarded by highly energized radicals and molecules projected ahead of the flame front. These are the environmental conditions that must be borne in mind. It is clear that they are such as would be expected to cause partial oxidation with the formation of intermediate oxygenated products, atoms and radicals, and it is the presence of one or more such bodies in critical concentrations which sensitizes the mixture and doubtless renders it prone to knock.

A certain amount of information, both direct and indirect, is available about the kinetics of these initial processes. There is no question, for example, but that pressure, temperature and time are important factors; and experiment has shown that the rate of sensitization may be accelerated or slowed down by the introduction of small quantities of promoters and inhibitors, respectively. These facts suggest that the high rate of combustion producing knock is due to a reaction of the branched chain type the chain-carriers being in all probability radicals derived from the thermal decomposition of some intermediate product formed in adequate concentration during the sensitizing period.[4]

A large number of inhibitors or anti-knocks is known, although only one, namely lead tetrethyl, has come into general use. Usually, though not invariably, they turn out to be volatile organo-metallic compounds which decompose to give metallic atoms and radicals, and, as Egerton has pointed out, the metal is always one that is capable of forming both a lower and a higher oxide. They have very little effect upon the speed of flame propagation or upon the setting up of true detonation, but they appear to act either by retarding the initial slow reaction or by limiting the number of reaction centres from which chains are set up.

From the point of view of the production of high-duty fuels (*i.e.*, fuels of high knock-rating), considerable interest attaches to the precise mechanism of the chain reactions which are responsible for knock, and a large

PLATE I (*a*).

GRAPHICAL PICTURE OF FLAME IN A MIXTURE OF $2CO + O_2$ IN A CLOSED TUBE 1·6 m. LONG IGNITED CENTRALLY.

[*To face p.* 138.

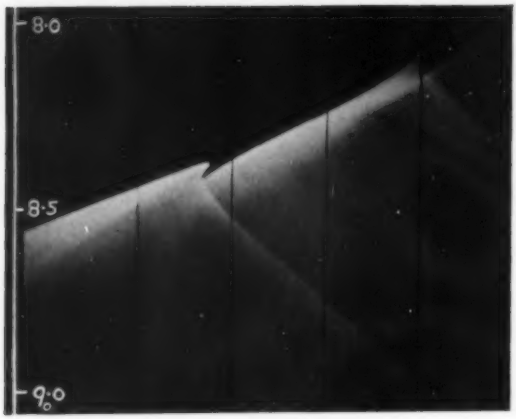

PLATE I (*b*).

DETONATION IN A CARBONIC OXIDE-OXYGEN MEDIUM.

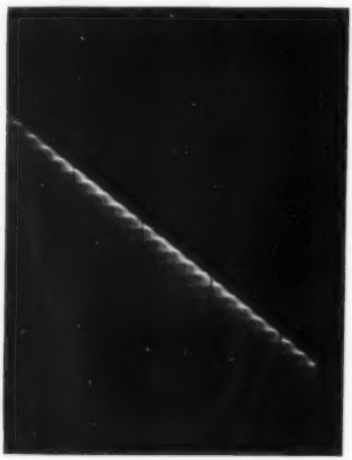

PLATE II (*a*).

THE SPINNING DETONATION WAVE IN A MIXTURE OF $2CO + O_2$ IN A TUBE 1·3 cm. DIAMETER. ROTATIONAL SPEED 44,000/sec. DETONATION SPEED 1760 m./sec.

PLATE II (*b*).

FLAME TRAVERSING AN ENGINE CYLINDER.

amount of work has been done in recent years in attempts to identify them. The problem is by no means an easy one. The slow combustion of any of the higher hydrocarbons leads to the formation of a very complex mixture containing aldehydes, alcohols, peroxidic bodies and acids in varying proportions, and the chain-carrier may be present amongst them in quantities so small as to defy detection by ordinary analytical methods. It is possible, however, to narrow the field. The dependence of knock on pressure, temperature and time suggests that the substance we are looking for must be relatively unstable under the working conditions and must be capable of breaking down to give active radicals which can act as chain-carriers. The fact that very small quantities of inhibitors (*e.g.*, 1 vol. of lead tetraethyl in 1300 vols. of fuel), are effective, indicates that it need be present only in low concentrations, and it is also reasonable to suppose that it would belong to the class of promoters and, when added to a fuel, would act as a powerful knock-inducer.

The substances which might be formed during slow oxidation and which conform to the above specification are alkyl-, alkyl hydrogen- and alcoxy-peroxides, aldehyde peroxides and peroxides of oxygenated ring compounds (*e.g.*, vinyl ether peroxide). Oxides of nitrogen which are also present are not pro-knocks. The presence of one or more organic peroxides in the exhaust gases of an engine has been demonstrated by Dumanois, Mondain-Monval and Quanquin,[5] and they have also been detected by Egerton, Smith and Ubbelohde [4] in the gases extracted from an engine just prior to knock.

On examining the "promoting" characteristics of typical members of each of the above classes of peroxide it is found that whilst olefine peroxides have only a slight effect, acetyl-, ethyl hydrogen- and diethyl-peroxides are powerful pro-knocks. The results as a whole suggest that only those peroxides are effective which decompose to give –OR and –OH radicals, the former most probably acting as chain-carriers. In this connection mention may be made of a recent detailed study of the decomposition of diethyl peroxide by Harris and Egerton.[6] They find that whilst the slow decomposition takes place according to a unimolecular law there is a certain critical pressure above which it becomes explosive; the stabilized products in the former case are acetaldehyde and ethyl alcohol and in the latter formaldehyde and ethane.

Having inferred something about the type of reaction which is or may be responsible for knock we may now attempt to find a correlation between the tendency to knock and the chemical reactivity of the fuel. It is known that all straight-chain paraffins, with the possible exception of methane, knock readily, whilst branched chain paraffins, olefines, diolefines and the simple aromatic hydrocarbons have comparatively high knock ratings. Generally speaking, the more compact the structure of the molecule the less likely is it to knock. Now, a convenient measure of the chemical reactivity of a fuel is its spontaneous ignition temperature, and the latter should, therefore, be closely related to the knock rating.

M. Prettre [7] has measured the ignition temperatures of a wide range of hydrocarbons at atmospheric and reduced pressures, and D. T. Townend [8] has investigated the relation between the spontaneous ignition temperatures and pressure up to pressures of about 15 atmospheres. We may refer to

the latter investigation, which is of particular significance in connection with the knock problem.

The spontaneous ignition temperatures over a wide range of pressures are conveniently measured by admitting the previously mixed fuel-gas medium into a heated vessel. Provided the temperature is sufficiently high there is usually a short delay period or "lag" during which no appreciable change of pressure occurs, followed by an abrupt increase in pressure, and ignition. The time-lags can be measured by means of a stop-watch and the ignition indicated by a suitable pressure gauge.

If we consider first some non-knocking fuels, such as methane, ethylene and benzene, we find that the ignition temperature falls progressively and

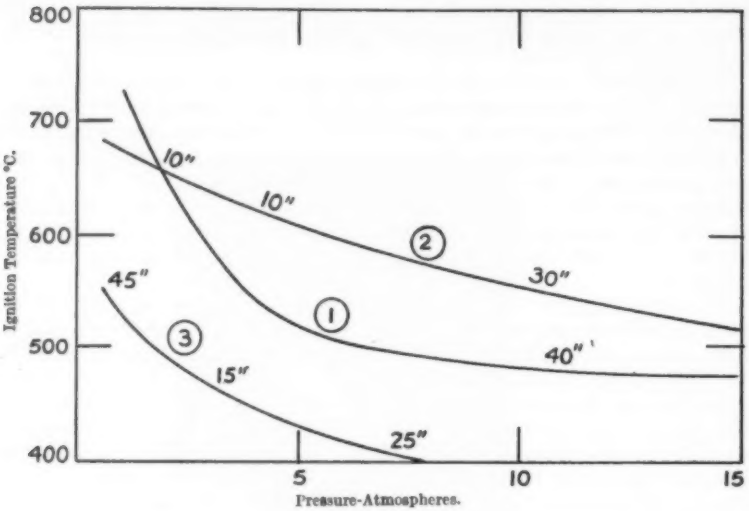

FIG. 1.

(1) 13% Methane.
(2) 1·36% Benzene.
(3) 10% Ethylene.

slowly with increase in pressure, as shown by the smooth curves in Fig. 1. The small figures adjacent to the curves indicate the time-lags before ignition. This is the type of relation that might be expected on the basis of the thermal definition of ignition and of a comparatively simple oxidation process, and needs no further comment. On comparing the results with the corresponding curves for some typical knocking fuels, namely *n*-octane and *n*-heptane, which have a low knock-rating, and *iso*-octane which has a high one (Fig. 2), we are at once struck by a marked difference in the character of the pressure effect.

In the case of the two normal paraffins the first effect of increase of pressure is to cause a very rapid fall in the ignition temperature and a corresponding decrease in the time-lags preceding ignition. Below about

400° C. there is a low-temperature system in which may be noted two pressure minima of ignition, and above 3 atmospheres ignition occurs at about 250° C., with progressively increasing time-lags. The shaded area to the left of the ignition curve marks the region in which "cool" flames are formed. The curve for *iso*-octane is somewhat similar, but there is only one pressure minimum in the low-temperature system occurring at a pressure of about 3 to 4 atmospheres higher than in the case of the normal paraffins. The minimum ignition temperature is also some 40° higher and the cool-flame region is more extensive.

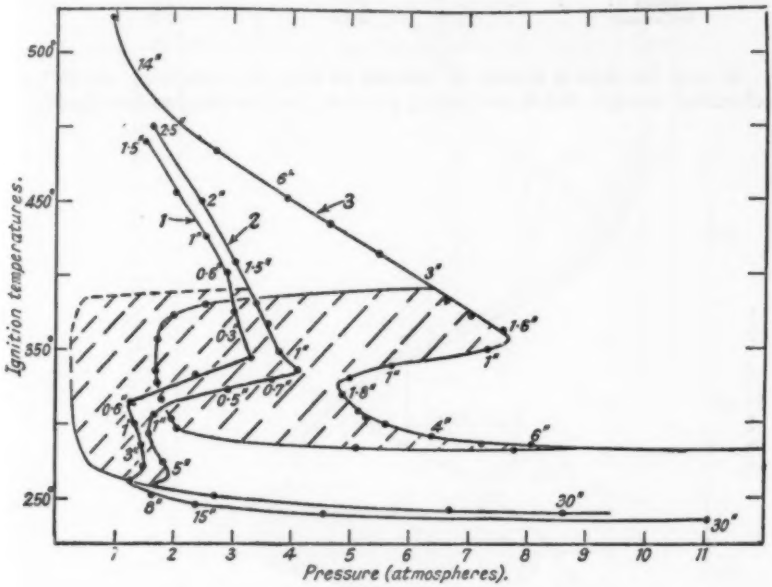

FIG. 2.

CORRESPONDING MIXTURES WITH AIR OF (1) *n*-OCTANE; (2) *n*-HEPTANE; (3) *iso*-OCTANE.

(Figures along curves denote time-lags (seconds)).

The conditions of temperature and pressure in an internal combustion engine are such as to suggest that knock is related to the reactivity in the low-temperature ignition system as determined by the curves in Fig. 2; and if we compare, as in Table I, the pressure minima read off from a series of such curves with the critical compression ratio of the fuels in question, as measured by a variable compression engine, we find that the fuels may be arranged in the same order.

In comparing these results it must be borne in mind that the minimum pressures relate to time-lags of the order of 1 sec. or more, whereas under engine conditions the time-lags would have to be of the order of a few thousandths of a second.

TABLE I.

A Comparison of the Minimum Ignition Pressures and the Critical Compression Ratios of a Number of Paraffin Fuels.

Fuel.	Minimum pressure, atms.	Critical compression ratio.
Methane	—	14·0
Propane	6·8	12·0
Butane	3·2	6·4
Pentane	2·2	3·8
Heptane	1·58	2·8
Octane	1·3	2·8
*iso*Octane	4·85	7·6

It now becomes a matter of interest to discover something about the chemical changes which are taking place in the low-temperature ignition

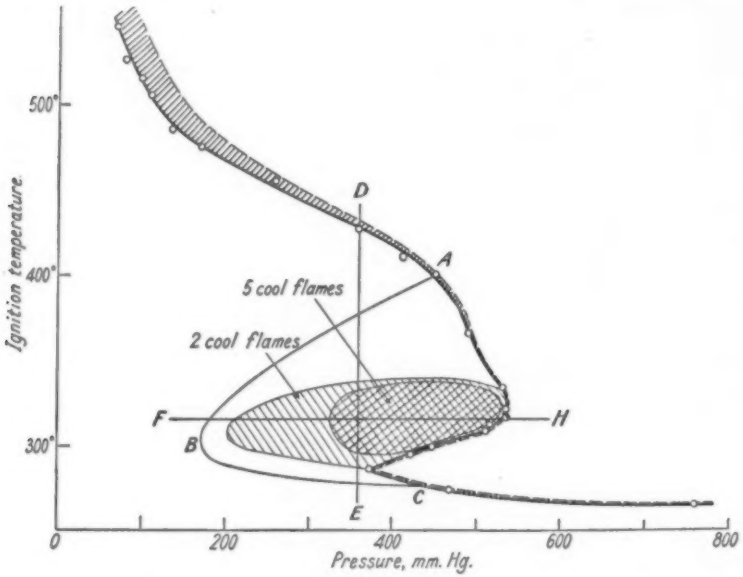

FIG. 3.

SPONTANEOUS IGNITION TEMPERATURES OF A $C_3H_8 + O_2$ MIXTURE.

system. Newitt and Thornes [9] have examined propane in detail and have made careful analyses of the products from combustion in the cool-flame region and in the slow-combustion zone immediately above it. The ignition temperature–pressure curve (Fig. 3) for an equimolecular propane–oxygen mixture resembles that for *iso*-octane, inasmuch as there is only one pressure minimum situated at about 0·5 atm. The cool-flame region is also well defined, and there are zones within it in which two or more separate cool flames traverse the medium at intervals of some 70 seconds,

each one being accompanied by a small pressure pulse. By carrying out the reaction in a quartz vessel it is possible to remove the vessel from the furnace in which it is being heated, and plunge it immediately into a freezing mixture, so as to arrest the reaction at any desired intermediate stage. In this way Newitt and Thornes have succeeded in following the course of the cool-flame- and slow-reactions in some detail.

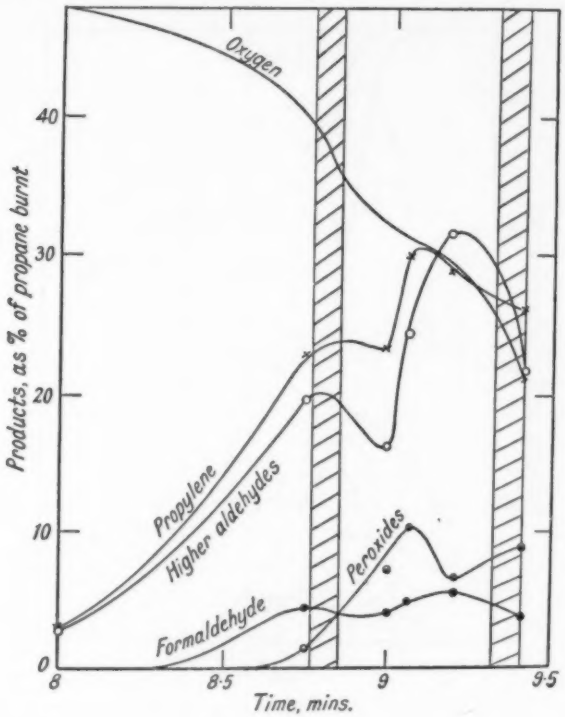

FIG. 4.

PRODUCTS FROM THE REACTION OF A $C_3H_8 + O_2$ MEDIUM AT 400 MM. AND 294°.

Attention may be drawn to two series of experiments carried out by them along the isobaric line *ED* (Fig. 3) in the cool-flame region and in the slow-combustion region immediately above it, respectively. The results are shown graphically in Figs. 4 and 5 and are summarized in Tables II and III.

Considering first the slow-combustion experiments, it will be seen that there is an induction or lag period of 10–15 secs. followed by a reaction period of 2·5 mins. during which intense luminescence is visible in the reaction vessel. In the gaseous products upwards of 30 per cent. of the carbon of the propane burnt appears as propylene at all stages of the reaction; both acetylene and methane are also present throughout, diminishing in quantity as the reaction approaches completion. Amongst the

liquid products peroxidic bodies and aldehydes appear in comparatively large quantities, the peroxides reaching a maximum when the reaction is proceeding with maximum velocity and representing no less than 19·9 per cent. of the carbon of the propane burnt. The higher aldehydes reach a maximum somewhat earlier, whilst formaldehyde, present at the commencement of the reaction to the extent of 4·6 per cent., gradually diminishes as reaction proceeds.

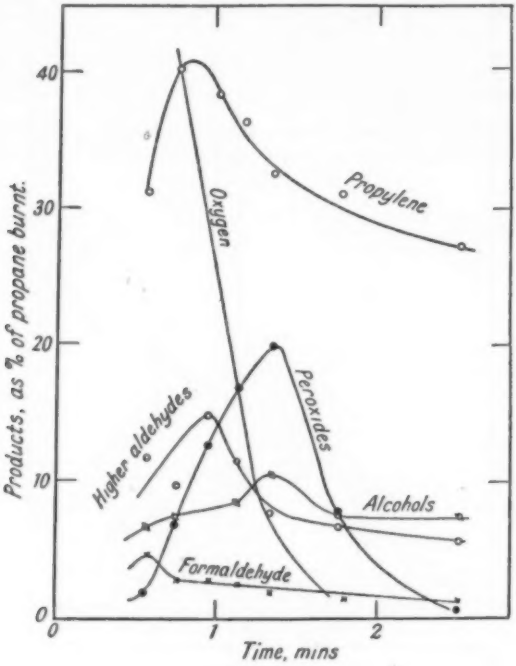

FIG. 5.

PRODUCTS FROM THE COMBUSTION OF A $C_3H_8 + O_2$ MEDIUM AT 360 MM. AND 400°.

Attempts to identify and isolate the peroxidic substances have not yet been successful. It seems probable that they are responsible for the ethyl alcohol which constitutes the greater part of the higher alcohols found in the cool-flame experiments. They are also decomposed by concentrated potassium hydroxide solution liberating a gas consisting of 80 parts of hydrogen to 20 parts of oxygen. These and other facts point to the presence of a mixture of peroxides containing an alkyl peroxide, a per-acid and/or hydrogen peroxide.

Turning now to the series of experiments in the cool-flame region, data are presented up to and including the passage of the second cool flame. The induction period for the reaction is 8 mins., the first cool flame is observed after 8·75 mins., the second after 9·4 mins., and reaction is complete

TABLE II.

Products from the Combustion of a $C_3H_8 + O_2$ *Mixture at 294° and 400 mm. Pressure.*

Duration of induction = 8 mins.; duration of reaction = 3 mins.

Expt. No.:	14.	15.	16.	17.	18.	19.
Time of sampling, in secs., from start	480	525	540	544	552	565
Oxygen content of sample, %	50·2	42·0	36·1	33·7	33·0	25·0
Products as percentages of the carbon of the propane burnt: C_3H_6	3·7	22·9	23·4	30·0	28·8	26·0
Products as percentages of the carbon of the propane burnt: CO	8·6	14·6	15·8	17·5	18·7	20·0
Products as percentages of the carbon of the propane burnt: CO_2	0·8	4·7	4·0	2·4	5·0	5·0
Acids	2·8	13·0	11·1	10·9	10·0	9·2
Higher aldehydes	3·0	19·6	16·1	24·3	31·6	19·6
Formaldehyde	nil	4·4	4·1	4·9	5·5	3·9
Peroxides	nil	1·4	6·2	8·2	6·9	8·8
Ratio O_2/C_3H_8 consumed	0·24	1·05	1·14	1·40	1·37	1·52

TABLE III.

Products from the Combustion of a $C_3H_8 + O_2$ Mixture at 400° and 360 mm. Pressure.

Duration of induction = 10–15 secs.; duration of reaction = 2 mins. 35 secs.

Expt. No.:	7.	8.	9.	10.	11.	12.	13.
Time of sampling, in secs., from start	33	44	57	68	80	105	150
Oxygen content of sample, %	47·5	39·0	30·0	18·0	6·85	1·8	1·9
Products as percentages of the carbon of the propane burnt: C_3H_8	6·3	4·7	3·1	2·1	2·3	2·3	2·6
C_2H_4	31·1	40·1	38·2	36·3	32·5	31·0	27·3
CO	15·3	16·6	22·4	25·5	30·3	33·2	35·4
CO_2	0·9	2·6	4·8	3·8	4·4	4·7	5·2
CH_4	8·4	6·8	5·7	5·0	3·9	3·5	5·7
Higher aldehydes	11·7	9·4	14·5	11·3	7·6	6·6	5·6
Formaldehyde	4·6	2·6	2·8	2·1	1·9	1·3	1·3
Peroxides	1·9	6·8	12·6	16·9	19·9	7·7	0·5
Methyl alcohol	nil	1·8	3·0	4·2	5·7	6·2	6·1
Higher alcohols	6·7	4·5	4·2	3·9	4·5	1·2	1·2
Ratio O_2/C_3H_8 consumed	0·71	1·26	1·55	1·72	1·93	1·92	1·89

after 11 mins. The chemical changes occurring during this reaction are rather complex, but it is possible to correlate them to some extent with the cool-flame phenomena. Thus the analytical figures show that higher aldehydes, acids, propylene and carbon monoxide are present in detectable quantities at the outset of the reaction, but no peroxides, or formaldehyde, and only a trace of carbon dioxide. The higher aldehydes rise to a maximum 45 secs. after reaction starts, and at this point the first cool flame is observed. During its passage through the reacting medium the percentage of higher aldehydes diminishes whilst peroxides and formaldehyde make their appearance; the rate of oxidation of the propane also increases. Shortly after the extinction of the cool flame, the peroxides reach a maximum and the higher aldehydes a minimum. A little later the aldehydes again increase rapidly to a second maximum, higher than the first, whilst the peroxides simultaneously decrease; the second cool-flame ignition then takes place and is accompanied by changes in the products similar to those noted with the first flame. The rate of formation of propylene appears to follow that of the higher aldehydes at all stages, whilst carbon monoxide increases fairly uniformly throughout.

On the basis of the above experiments it may be concluded that a necessary condition for cool-flame inflammation and for ignition in the lower region is a critical high concentration of higher aldehydes. During the passage of the flame the aldehydes are further oxidized to give aldehyde peroxides and/or per-acids and ultimately formaldehyde. The reactions by which propylene and the higher aldehydes are formed also appear to be related.

Now although the conditions in these experiments differ in some material respects from those in an engine, particularly as regards the time factor, there is reason to suppose that the mechanism of the combustion is essentially the same in the two cases. It may, indeed, be assumed that the sensitized mixture in which knock originates is one having a composition not very different from that of a mixture in which cool-flame inflammation is about to take place. And if such a mixture were ignited by an external source there is little doubt but that a rapid combustion closely resembling that associated with knock would occur.

The products of the combustion of all the higher hydrocarbon fuels including branched-chain paraffins and olefines are similar to those of propane in the sense that aldehydes, alcohols, peroxidic bodies and acids are always formed. The proportions in which they survive at any particular stage of the combustion, however, varies from fuel to fuel and depends upon temperature and pressure; and it is this circumstance perhaps more than any other which constitutes the difference between a low and high knock rating fuel. It will be recalled, for example, that although a striking similarity exists between the ignition temperature–pressure curves for *n*-octane and *iso*-octane a higher pressure is always required to affect the ignition of the latter at any given temperature in the ignition range. This may be interpreted as indicating that a higher pressure is required to stabilize the initial products so that concentrations adequate to initiate cool flames may be built up.

The same considerations apply to the knock ratings of some of the more recently tested high-duty fuels such as acetone and di*iso*propyl ether;[8] in

both cases the pressures required to promote ignition in the low-temperature range are comparatively high. In the case of mixed fuels, as for example the petrols obtained by the hydrogenation of coal, the combustion characteristics depend largely upon their straight chain- and branched-paraffin, naphthene and volatile aromatic hydrocarbon content; and methods of controlling the relative proportions of these constituents by vapour phase hydrogenation in the presence of selective catalysts have recently been discovered and put into operation for the production of high-duty fuel.

It would be incorrect to assume that anything like finality had been reached in the development of high-duty fuels and of anti-knocks. During the past ten years there has been a steady improvement in the knock ratings of commercial fuels, and a number of new fuels have been introduced to meet the increasingly stringent demands of engine designers. In this connection some of the researches I have described or referred to are proving of considerable utility, particularly in indicating directions in which further advances may profitably be made. Finally, I think it will be admitted that, although the precise reactions responsible for knock and the inhibiting effect of anti-knocks have not yet been identified, the general character of the mechanisms involved are well established.

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[7] Prettre, *Ann. Comb. liq.*, 1931, **6**, 7, 269, 533 *et seq.*
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THE COMPOSITION OF SOME ROUMANIAN STRAIGHT-RUN GASOLINES.*

By C. D. Nenitzescu and A. Constantinescu.

Synopsis.

In the present paper the composition of some Roumanian straight-run gasolines is studied, not so much from the point of view of the identification or estimation of certain individual hydrocarbons occurring in these gasolines, but chiefly to obtain a general idea of the main classes of hydrocarbons occurring therein. The basis of modern research work dealing with the composition of gasoline is a distillation process, using high-efficiency apparatus, which has lately been greatly improved.[12] This method has been employed. The gasolines were distilled in a similar apparatus, and the narrow fractions obtained were then analysed by the most convenient methods, with a view to establishing the percentage of aromatic, naphthenic, normal paraffin and branched paraffin hydrocarbons occurring therein.

Fractionating Column.

For the construction of this apparatus we utilized the information given by M. R. Fenske and co-workers.[3] The height of the packed section was 6 m., whilst the total height of the distilling apparatus was 7 m. The column consisted of a brass cylinder which was packed with single-turn rings of 4 mm. diameter made of 0·5 mm. brass wire.

The still employed in our experiments (See Plate 1) was a vertical cylinder, 220 mm. diameter, 330 mm. high, in which 8·5–9 litres of liquid could be distilled. In order to test the efficiency of the apparatus, a smaller still of 1·5 litres capacity was used.

At the top of the column was fitted a total reflux condenser which could be connected by means of a small stopcock with the product line. The distilled product was collected in a graduated burette of 100-ml. capacity provided with two glass condensers.

In order to obtain a distillation under adiabatic conditions, the column was insulated with a mixture composed of asbestos and magnesium oxide over a layer of 10 cm. About the middle of this layer—*i.e.*, at a distance of 35 mm. from the column—a chrome-nickel resistance belt 0·1 mm. thick and 5 mm. wide was fitted. The column was wound in three separate sections, 2 m. long, each being controlled by a variable resistance and an amperimeter. By this method the column could be maintained, as desired, at a temperature equal to, or slightly below, that of the vapour phase. The same insulating and heating system was used for the still.

In each of the three heated sections, as well as on the top of the column, a Hartmann and Braun resistance thermometer was fitted, with which temperatures within 50–150° C. can be measured with a precision of 0·1° C. The boiling points of the fractions were measured with a thermometer fitted on the top of the column.

* Paper received September 27th, 1938.

Temperatures are read and controlled from a control-panel containing also the collecting burette for the products.

The actual volume of the packing material is 390 ml. The free space amounts, therefore, to 79·3 per cent. The total surface of the packing is approximately 3 sq. m., and the hold-up, which was determined as usual with kerosine, amounts to 78 ml.

During distillation a superpressure of 30–40 mm. Hg was observed, which was measured with a manometer fitted on the above-mentioned control-panel.

The column was tested under total reflux with a binary mixture consisting of *n*-heptane and methyl*cyclo*hexane,[4] the former being derived from Jeffreys pine and having the following constants: $D = 0{\cdot}6839$ and $n = 1{\cdot}3878$, whereas the boiling point determined with our column (at a pressure of 750 mm.) was 98·0° C. The methyl*cyclo*hexane used was purified by us in the usual manner. With our column we found a boiling point of 103·3° C. and a refractive index $n = 1{\cdot}1232$.

The efficiency test was carried out by distilling under total reflux a mixture of 2·73 mols. of each of the above-mentioned hydrocarbons. When equilibrium was reached, which took about 1 hour, two samples, a few millilitres each, were withdrawn from the top of the column as well as from the still. At this moment the temperature at the top of the column was 98° C.

The refractive index of the samples was determined at the temperature of 20 + 0·05° C. by means of a Pulfrich refractometer provided with a Höpler ultra-thermostat, which was also used for the other experiments in the present study.

For the sample taken on the top of the column we found a refractive index of $n = 1{\cdot}38887$, which, according to the tables published by E. C. Bromiley and D. Quiggle,[5] corresponds to 96·5 ml. per cent. *n*-heptane; for the sample taken from the still we found a refractive index of $n = 1{\cdot}40999$, corresponding to 33·7 mols. per cent. *n*-heptane.

We applied the formula of M. R. Fenske, C. O. Tonberg and D. Quiggle[6]

$$\frac{XnA}{XnB} = (\alpha^{n-1})\frac{X1A}{X1B}$$

where $\frac{X1A}{X1B}$ = molar ratio of *n*-heptane (*A*) to methyl*cyclo*hexane (*B*) on any plate, $\frac{XnA}{XnB}$ = molar ratio of *A* to *B* on any plate removed from the first; and n = number of perfect plates required for separation. α = the relative volatility, was taken from Beatty and G. Calingaert's paper[7] as equal to 1·07.

By means of the above formula we found that $n = 60$, corresponding to the theoretical number of plates of our column. Next, according to the following equation, we have :

$$H.E.P.T. = \frac{600}{60-1} = 10{\cdot}2 \text{ cm.}$$

After taking the two samples under total reflux, the product line was opened and the liquid distilled at the rate of approximately 0·8 ml. per

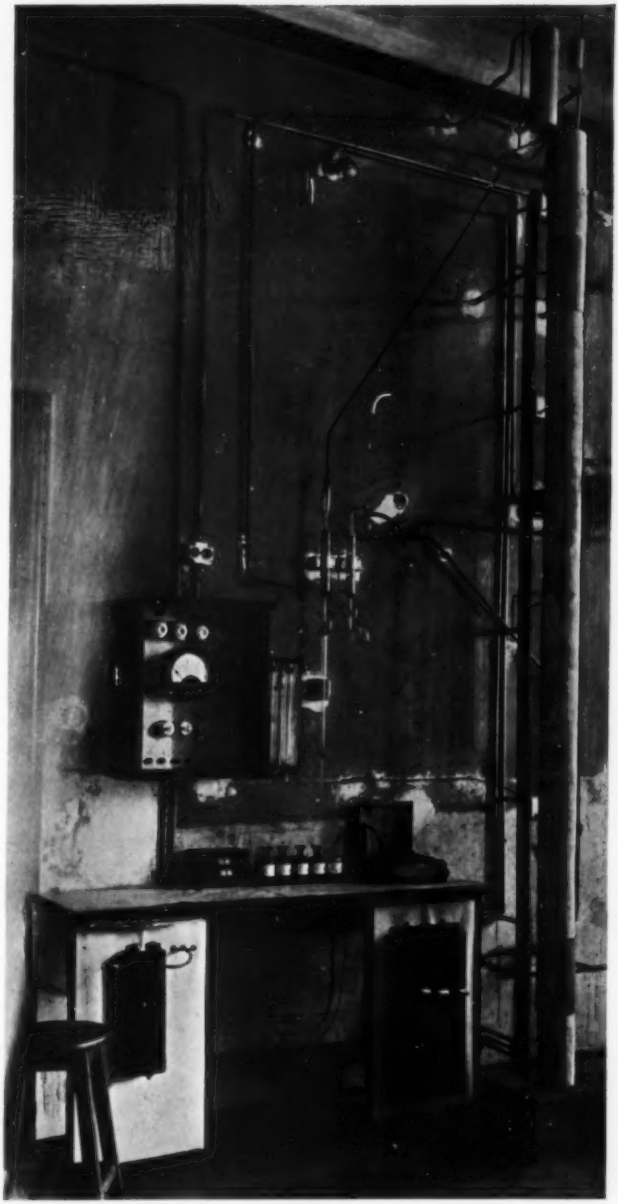


PLATE I.

FRACTIONATING COLUMN USED IN THE EXPERIMENTS.

[*To face p.* 150.

minute. So as not to complicate the apparatus, we discontinued determining the reflux ratio which, however, was later estimated at approximately 1 : 20–30. Twenty-six fractions, 20 ml. each, were collected, and the

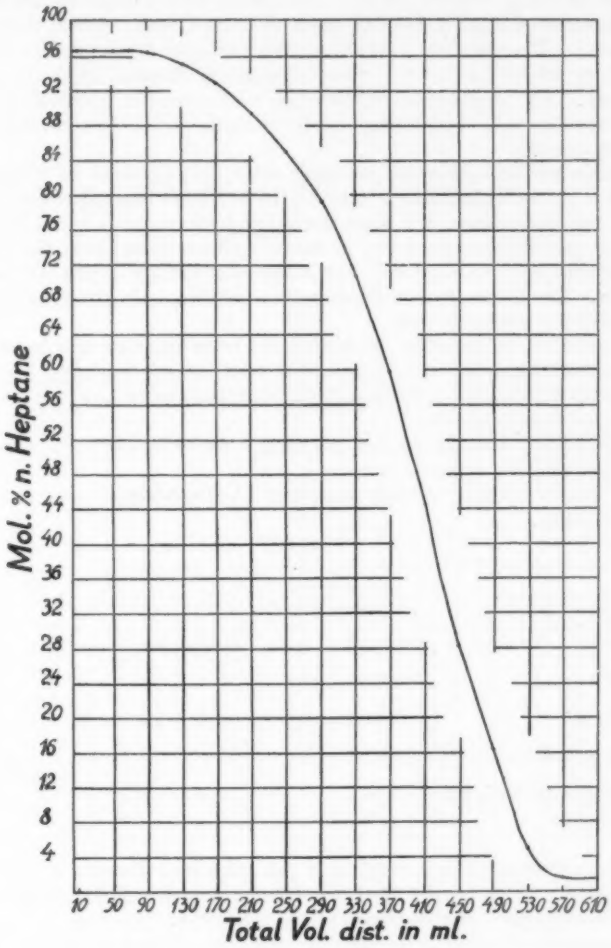


FIG. 1.

distillation process was then interrupted when the boiling point of the fractions attained 100·3° C. The residue amounted to 102 ml. The composition of each fraction was then determined by the above-mentioned method.

Analysed Gasolines.

In the present study three gasolines were examined—namely, one gasoline from a Bucşani paraffinic crude (Concordia Company), a gasoline from Merişor asphaltic crude (I.R.D.P. Company) and a high-octane-number gasoline selected from a Dacian crude of Gura Ocniţei oilfield (Concordia Company). The first two gasolines were prepared from crude oil in our laboratory, whilst the last one was placed at our disposal by the Concordia Company. All the gasolines were shaken with a sodium hydroxide solution to remove any acid which they might contain, and were then dried over metallic sodium.

The Merişor and Bucşani gasolines were first distilled in the column as such. As we found, however, that the aromatic hydrocarbons distilled over a too wide range, and thus the other hydrocarbons could be drawn together, we first removed the aromatic hydrocarbons from the Bucşani gasoline and then distilled it in our column, obtaining in this way sharper maxima. The Gura Ocniţei gasoline was distilled only after the aromatic hydrocarbons were removed.

The removal of aromatic hydrocarbons from gasoline was carried out by repeated shaking with concentrated and fuming sulphuric acid, until the presence of aromatic hydrocarbons could no longer be detected by the usual analytical methods.

All gasolines, as well as the fractions and residues, were completely saturated towards bromine water.

The characteristics of the gasolines thus obtained are shown in Table I below.

Table I.

Gasoline of :	$D_{15°}$	Ar, % vol.	O.N.	Engler Distillation.
Merişor	0·7362	6·5	63·0	I.B.P. 60° C.; 40·5% at 100° C.; F.B.P. 173° C.
Bucşani	0·7365	10·8	51·5	I.B.P. 68° C.; 29% at 100° C.; F.B.P. 182° C.
Gura-Ocniţei . . .	0·7435	7·5	76·0	I.B.P. 53° C.; 53% at 100° C.; F.B.P. 136° C.
Bucşani (aromatic-free) .	0·7262	0	48·0	I.B.P. 68° C.; 26·5% at 100° C.; F.B.P. 185° C.
Gura-Ocniţei (aromatic-free)	0·7350	0	75·0	I.B.P. 53° C.; 50% at 100° C.; F.B.P. 140° C.

The gasolines were then distilled in the column at the rate of 0·7–0·8 ml. per minute. Eight to nine litres were distilled, and a complete distillation lasted 8–10 days. The distillation was interrupted for only a very short time, sometimes being kept working continuously for 50 hours. During the interruptions the heat of the column was kept constant, whilst the heating of the still was interrupted. At each new start the column was worked under total reflux until a perfect equilibrium was established.

In order to avoid auto-oxidation, a small quantity of diphenylamine was added to the gasoline.

During the distillation process we collected first a top fraction up to 50° C., then 100 fractions, each distilling at intervals of 1° C., with the only

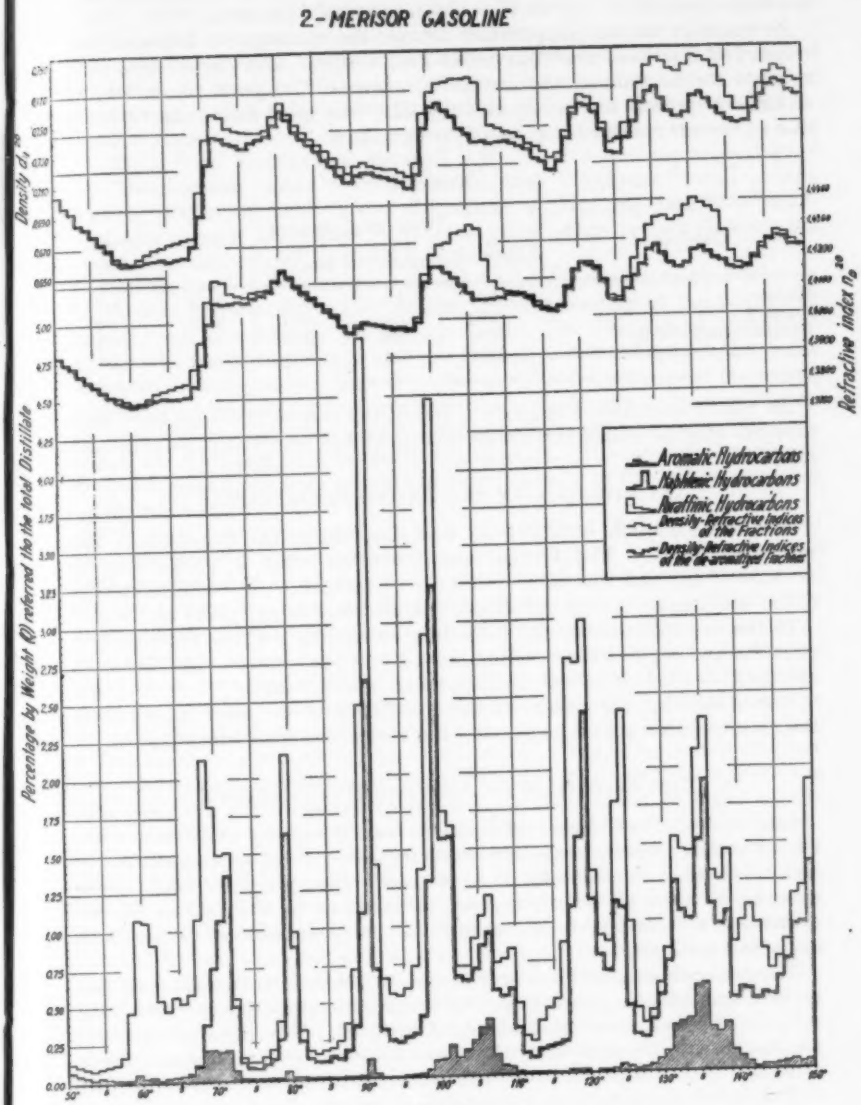

2-MERISOR GASOLINE
Density d_4^{20}
Refractive index n_D^{20}
Percentage by Weight (%) referred the the total Distillate
Aromatic Hydrocarbons
Naphtenic Hydrocarbons
Paraffinic Hydrocarbons
Density-Refractive Indices of the Fractions
Density-Refractive Indices of the de-aromatized Fractions

FIG. 2.

exception of the "Gura Ocniței" gasoline, where the distillation was interrupted at 123·5° C.; we thus obtained only 74 fractions.

In order to obtain comparative figures, the measured volumes of the respective fractions were converted into weights, and these were then referred to the bulk of the distilled fractions. The gross results of the distillation process are shown in Table II. The great losses observed are due to the uncondensable gases of the gasolines.

TABLE II.

Gasoline of:	Ml.	Fractions below 50° C.	Sum of Distillate, %.	Residue, %.	Losses, %.
Merișor	9200	3·5	63·8	24·5	8·2
Bucșani	8000	2·6	56·5	30·0	10·9
Bucșani (aromatic-free) . .	8000	2·6	55·5	30·7	11·2
Gura-Ocniței (aromatic-free) .	8000	5·1	69·9	12·3	12·7

As will be seen from Figs. 2, 3, 4 and 5, the various fractions have very unequal weights, whilst the distillation curve has very sharp maxima and minima.

INVESTIGATION OF THE FRACTIONS OBTAINED.

For each fraction obtained we first determined the density and the refractive index. The former was measured with a picnometer at 20 + 0·05° C., and was then reduced to water at 4° C. The refractive index was measured with a Pulfrich refractometer as explained above.

In the case of gasolines still containing aromatics, we then proceeded to estimate and remove these hydrocarbons. In the aromatic-free fractions obtained we then estimated the naphthenes and paraffins.

Before showing the results of these estimations, we must state briefly the reasons which guided us to select the various methods adopted.

THE ESTIMATION OF AROMATIC HYDROCARBONS.

One of the three classes of hydrocarbons occurring in gasoline—*i.e.* the aromatic hydrocarbons—are characterized by a far greater reactivity and by physical constants much more specific than the other two. However, as we shall see below, no method exists up to the present for the estimation of these hydrocarbons with the precision required by the usual analytical methods.

The methods described in literature for the estimation of aromatic hydrocarbons occurring in gasoline may be divided into three groups: chemical methods, physical methods and mixed methods. The chemical methods use as reagents the sulphuric–nitric acid mixture, concentrated or fuming sulphuric acid or formaldehyde in the presence of sulphuric acid.

This last method is not precise enough to be considered.

Numerous methods utilize the sulphuric–nitric acid mixture.[8] The methods of Hess and of Egloff and Morrell are sufficiently known, but from the studies of Riesenfeld and Bandte[9] and those of Faragher, Morrell and Levine[10] it follows that these methods, in spite of the fact that they give

satisfactory results, present no advantage whatever as compared with those which use 98 per cent. acid. The use of fuming sulphuric acid in various concentrations is to be found in the older literature; [11] this reagent, however, readily attacks the hydrocarbons of the other classes occurring in gasoline. Zelinsky,[12] for the first time, uses acids of lower concentration than 100 per cent. for the estimation of aromatic and ethylenic hydrocarbons. An acid of approximately 98 per cent. was then suggested by Thole,[13] by Tizzard and Marshall,[14] by Dănăilă and his co-workers,[15] by Morgan and Soule,[16] by Sachanen and Wirabianz [17] and others. Finally, we must mention the suggestions of Manning and Shepherd,[20] and also those of Katwinkel,[21] who add silver sulphate and phosphorous pentoxide, respectively, to the sulphuric acid.

Dănăilă and his co-workers use a sulphurimeter similar to that used by Krämer and Böettcher, which enables a direct reading of the volume of gasoline not absorbed by the sulphuric acid. In this manner the above method becomes very rapid and convenient. The estimation is carried out with 10-ml. gasoline, and the tube of the sulphurimeter is graduated in 0·1 ml., so that one division corresponds to 1 per cent. aromatics. Later on the authors have suggested sulphurimeters graduated in 0·05 ml., which are filled and read in special thermostats. We shall see below that these improvements were not of much use, because of the lack of precision of the method.

The main criticism brought against this method of directly estimating the volume of aromatic hydrocarbons by absorption in sulphuric acid is that the saturated hydrocarbons are also absorbed by the sulphuric acid: thus we have known for a long time that *n*-octane is absorbed by 97 per cent. sulphuric acid, under certain mild conditions, in a proportion of 2 per cent. The same conclusion was reached by Cazimir,[19] working on aromatic-free gasoline with an acid of 98·3 per cent. The values found for the aromatic hydrocarbons are, therefore, greater than the real ones. Similar remarks are made by Ormandy and Craven.[23]

Taking these conclusions into consideration, it would seem that the best way to estimate aromatic hydrocarbons in gasoline would be a combined method: the determination of the density and refractive index of the respective fraction, the removal of aromatics from the fraction with sulphuric acid under mild conditions and a final determination of the density and refractive index. The percentage of aromatics is then calculated by means of Thole's [13] formula, using the density, or with Hoyte's [22] formula, which uses the refractive index. This method is connected with another source of error which escapes control—namely, when aromatics are mixed with saturated hydrocarbons a contraction of volume takes place, depending on the nature of the saturated hydrocarbons present, so that the additivity rule on which the above calculation is based is no longer valid.[24]

With regard to the physical methods applied or suggested for the estimation of aromatics in gasoline, the only one worth mentioning is the well-known "aniline-point" method. This method, which may be of service when it is only a question of estimating aromatics in large fractions of gasoline, was not sufficiently precise for our purpose, because, as Tilitschejew and Dumskaja [24] have shown, the coefficients vary with the concentration of the aromatic hydrocarbons as well as with the nature and chemical

composition of the non-aromatic portion of the sample. The coefficients mentioned in literature which serve to determine the percentage of aromatics (1·15 for a fraction boiling at 60·95° C.; 1·20 for a fraction of boiling point 25–122° C.) were determined for wide gasoline fractions containing a large number of compounds. The application of these coefficients to narrow fractions such as ours would have introduced an element of inaccuracy which we do not believe to be less than that encountered in the 98 per cent. sulphuric absorption method. It is true that this difficulty might be overcome in a large measure by determining some of the aniline points for each of our aromatic-free fractions, by adding the pure aromatic hydrocarbon and by determining the aniline point afterwards. This procedure would, however, have greatly complicated the method, and would have lessened much of its practical value. Therefore, we chose for the estimation of aromatic hydrocarbons in our narrow distilling fractions the method of Dănăilă and his co-workers, which is, likewise, very expedient.

With regard to the errors which may be made by the application of Dănăilă's method, we have observed the following : if aromatic-free gasoline fractions are treated exactly according to this method with a 98 per cent. acid, we observed an absorption of approximately 1·0–1·2 per cent. In the case of the Bucşani gasoline, the 92–93° C. fraction had, before and after treatment with sulphuric acid according to Dănăilă's method, the same density and refractive index as before. Consequently this fraction does not contain any aromatic hydrocarbons. It still shows, however, an absorption of 1·2 per cent. in sulphuric acid when treated by Dănăilă's method, even when avoiding with care any heating during the first minutes of the shaking. This applies also to fractions 93–94, 94–95, 118–119 and 119–120. It follows, therefore, that all the values for the aromatic hydrocarbons obtained by us are about 1 per cent. higher than the real figures. As these remarks were made only at the conclusion of this study, we did not attempt to make any revision in this sense.

Estimation of Naphthenic Hydrocarbons when Mixed with Paraffins.

After estimating the aromatic hydrocarbons by Dănăilă's method, the sulphurimeter is emptied in a separating funnel, the acid removed and the fractions are washed and dried over metallic sodium. A series of fractions were obtained in this way containing only paraffins and naphthenes. The fractions obtained by previously distilling the aromatic-free benzines showed the same composition. The specific gravity and refractive index were then determined for these fractions by the methods outlined above.

For the estimation of the amount of naphthenes contained in these fractions, we had two methods at our disposal : that of the aniline point and that of specific refraction.

The first-mentioned method is too inaccurate to be considered for narrow fractions. Data published in various papers on the subject [14, 17, 23, 25] show that some paraffins have very similar boiling points, yet widely differing aniline points.

On the other hand, the classic method of specific refraction is very useful in this case, as the substances concerned may be mixed together without

contraction of volume, so that the additivity rule becomes applicable in its entirety. The specific refraction of the fraction may be calculated on the basis of the specific gravity and the refractive index by the well-known Lorentz–Lorenz formula, and is a constant quantity independent of temperature. The specific gravity and the refractive index may be determined with great accuracy, and specific refraction is, therefore, in a large measure free from experimental error.

The specific refraction of the mixture and that of the paraffins and naphthenes contained in this mixture being known, the percentage naphthenes by weight may be obtained from the formula :

$$\text{Naph. } \% = 100\frac{Rp-Rf}{Rp-Rn}$$

in which *Rf*, *Rp* and *Rn* are the specific refractions of the analysed fraction, and of the paraffins and naphthenes contained in the respective fraction.

The specific refractions of the pure substances may be calculated from the molecular refraction by dividing same by the molecular weight of the respective substance. Likewise, molecular refraction may be calculated from the atomic increments,[27] which, being determined for a very large number of substances, offer an incomparable degree of accuracy compared with other constants, such as, for example, critical solution temperature in aniline.

Gasolines with a boiling point up to 200° C. contain only monocyclic naphthenes with a general formula C_nH_{2n}. These have a constant specific refraction equal to 0·3296, independent of molecular weight.

Thus, two out of the three factors of the above formula may be determined with great accuracy. Unfortunately, the specific refraction of paraffins contained in a wide fraction of gasoline cannot be given with the same precision, as in the case of paraffins specific refraction varies with molecular weight. This is, doubtless, the reason why this method is not more widespread to-day.*

However, in the case of narrow and well-separated fractions, such as those with which we have worked, the specific refraction of the paraffins may be indicated with sufficient accuracy. Thus, if we examine the distillation curve of the Merişor gasoline, we see that it is composed of a series of maxima separated by a series of well-defined minima. Minima are thus encountered at 75° C., at 103° C., and again at 128° C. Taking the boiling points of various paraffins into consideration, we may infer that in the interval between 50° and 75° C. only isomeric hexanes are encountered, between 75° and 103° C. only isomeric heptanes and from the latter temperature to 150° C. only isomeric nonanes.

In the case of the other distilled gasolines, we encounter minima at the same temperatures, so that the intervals admitted above for the distillation

* Marder,[24] in a comprehensive study concerning the analysis of gasoline by physical methods, attempts to avoid this disadvantage by measuring the average molecular weight of gasolines and determining by a graphic method the mean specific refraction corresponding to the mean molecular weight. This method naturally loses all accuracy with fractions boiling over somewhat wider limits. In calculating the percentage of naphthenes, Marder uses a formula similar to that shown above, the percentage of naphthenes being, however, expressed in volumes.

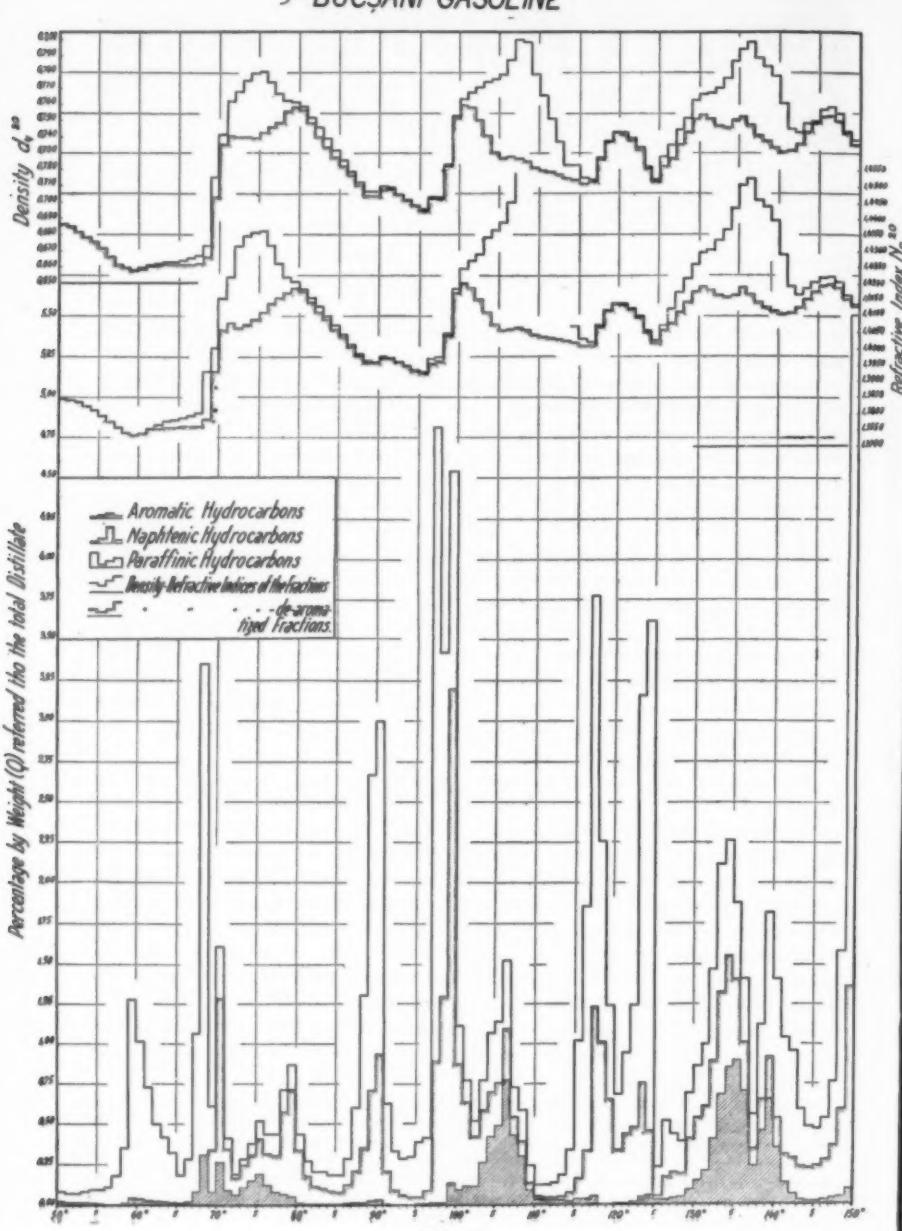

3-BUCȘANI GASOLINE
Density d_4^{20}
Refractive Index n_D^{20}
Percentage by Weight (Q) referred tho the total Distillate
Aromatic Hydrocarbons
Naphtenic Hydrocarbons
Paraffinic Hydrocarbons
Density-Refractive Indices of the fractions
de-aromatized Fractions

FIG. 3.

of hexanes, heptanes, octanes and nonanes are the same for all gasolines, if the distillation column has sufficient separating power.

Consequently the above-mentioned formula may be applied by making use of the specific refractions of paraffins calculated from Eisenlohr's increments, as shown in Table III.

TABLE III.

Specific Refractions of Paraffins (Rp) Contained in Various Gasoline Fractions.

Fractions.	C_nH_{2n+2}.	*Rp.*
50–75	C_6H_{14}	0·3463
75–103	C_7H_{16}	0·3450
103–128	C_8H_{18}	0·3431
128–150	C_9H_{20}	0·3416

On examining the distillation curves of the gasolines, and by taking into consideration the boiling points of pure paraffins,[28] it is apparent that it is not possible to find appreciable quantities of hexanes in the maxima above 75° C. and of heptanes below that temperature. Similarly, heptanes cannot be found in the maxima above 103° C. nor octanes within the maxima below that temperature. Uncertainty exists only with regard to the nature of the paraffins in the fractions corresponding to the minima of the curve—for example, fractions 74–76, 102–104, etc. The paraffins in these fractions may be hexanes mixed with heptanes, heptanes with octanes, etc. As these fractions, however, represent only a small quantitative percentage of the total gasoline, the error resulting from the uncertainty regarding the composition of these fractions, and from arbitrary choice of the limits shown in Table III, becomes negligible.

EXAMPLES OF CALCULATION.

As shown above, the percentage of hydrocarbons may be determined by the method of specific refractions *by weight*, and we were thus forced to compute all our results obtained in terms of weight. For this reason we consider as specific gravity of aromatics distilling up to 86° C. (benzene)= 0·8736, of those distilling up to 120° (toluene) = 0·8660, and of those distilling up to 143° (ethyl benzene, *o*-, *m*-, and *p*-xylenes) = 0·8680 whilst those distilling between 133° and 150° C. (in which *o*-xylene predominates)= 0·8790.

Likewise, the total weight and mean specific gravity of the sum total of distilled fractions were calculated on the basis of the volume and specific gravity of each fraction.

Gasoline of:	Total Volume, ml.	Total Weight, gms.	D_4^{20}, average.
Merişor	5871	4357	0·7421
Bucşani	4519	3318	0·7343
Bucşani (aromatic-free) . . .	4438	3206	0·7224
Jura-Ocniţei (aromatic-free) . . .	5589	4135	0·7398

We give below as an example the calculation for the fraction 97–98° C. of Bucşani gasoline. It had the following constants: $D_4^{20} = 0{\cdot}7033$; $n_D^{20} = 1{\cdot}39673$ and Ar per cent. vol. = 1·3 per cent. After removal of aromatics $D_4^{20} = 0{\cdot}7027$ and $n_D^{20} = 1{\cdot}39624$. The specific refraction of the aromatic-free fraction is therefore 0·3421.

The percentage volume of aromatics were transformed into weight as follows:

$$A = \frac{\text{aromatics \% vol.} \times \text{density of aromatics}}{\text{specific gravity of original fraction}} = \frac{1{\cdot}3 \times 0{\cdot}8660}{0{\cdot}7033} = 1{\cdot}6 \text{ per cent.}$$

The percentage, by weight, of naphthenes in the aromatic-free fraction is, according to the above reasoning:

$$N_1 = 100\frac{Rp - Rf}{Rp - Rn} = 100\frac{0{\cdot}3450 - 0{\cdot}3421}{0{\cdot}3450 - 0{\cdot}3296} = 18{\cdot}8 \text{ per cent.}$$

The percentage, by weight, of paraffins in the same fraction amounts to:

$$P_1 = 100 - 18{\cdot}8 = 81{\cdot}2.$$

Referring these figures to the original fraction we have:

$$N = \frac{(100 - A) \times N_1}{100} = \frac{(100 - 1{\cdot}6) \times 18{\cdot}8}{100} = 18{\cdot}5$$

$$P = \frac{(100 - A) \times P_1}{100} = \frac{(100 - 1{\cdot}6) \times 81{\cdot}2}{100} = 79{\cdot}9$$

This type of calculation was used throughout for each separate fraction. The calculation is, naturally, simplified in the case of fractions obtained from aromatic-free gasoline.

In order to have comparative figures, the results were referred to the total fractions distilling between 50° and 150° C. as shown above, in the following manner:

Fraction 97–98 of Bucşani gasoline had a vol. = 227 ml. and D = 0·7033. The weight of the fraction is 227 × 0·7033 = 159·6 gm. Referred to the total distillate, this fraction represents:

$$Q = 100\frac{159{\cdot}6}{3318} = 4{\cdot}812 \text{ per cent.}$$

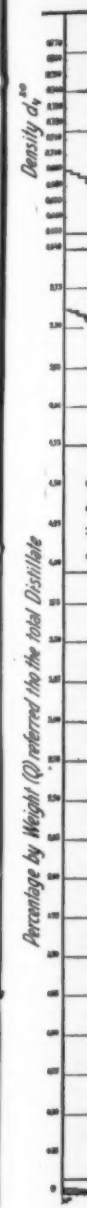
If we represent the contents in aromatic, paraffinic and naphthenic hydrocarbons referred to the total distillate as Q_A, Q_P and Q_N, we obtain:

$$Q_A = \frac{159{\cdot}6 \times 1{\cdot}6}{3318} = 0{\cdot}077 \text{ per cent.}$$

$$Q_P = \frac{159{\cdot}6 \times 79{\cdot}9}{3318} = 3{\cdot}845 \text{ per cent.}$$

$$Q_N = \frac{159{\cdot}6 \times 18{\cdot}5}{3318} = 0{\cdot}890 \text{ per cent.}$$

$$Q = 0{\cdot}077 + 3{\cdot}875 + 0{\cdot}890 = 4{\cdot}812.$$



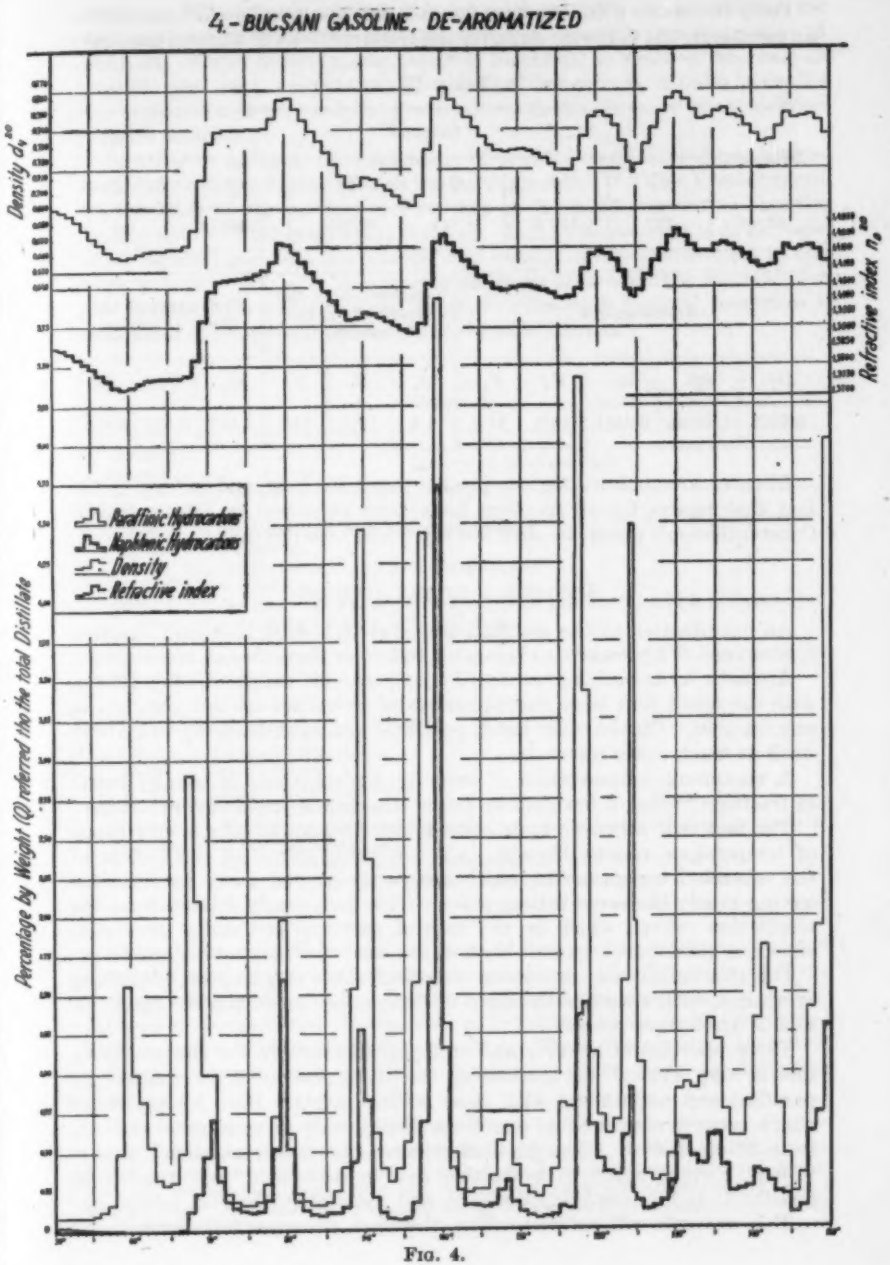

4.-BUCȘANI GASOLINE, DE-AROMATIZED
Density d_4^{20}
Refractive index n_D^{20}
Percentage by Weight (%) referred the the total Distillate
Paraffinic Hydrocarbons
Naphtenic Hydrocarbons
Density
Refractive index

FIG. 4.

From the above it follows that for each fraction a table has been drawn up containing the following data, for the fraction 97–98 of Bucşani gasoline:

TABLE IV.

Fraction.	Original.					
	ml.	Gm.	Q.	D_4^{20}.	n_D^{20}.	Ar, % vol.
97–98	227	159·6	4·812	0·7033	1·39673	1·3

Aromatic-free:					Composition of the Fraction, % by weight.			Percentage of the Total Distillate.		
D_4^{20}.	n_D^{20}.	R_f.	N_1.	P_1.	A.	N.	P.	Q_A.	Q_N.	Q_P.
0·7027	1·39624	0·3421	18·8	81·2	1·6	18·5	79·9	0·077	0·890	3·845

In order to avoid making our present paper too long, and in view of the fact that figures for all fractions have been published *in extenso* in Mr. Constantinescu's thesis, we shall not reproduce here the tables.

DISCUSSION OF THE RESULTS.

An examination of the distillation curves will show that each fraction is composed of hydrocarbons belonging to two or three classes, respectively.

Aromatic hydrocarbons are distributed over wide ranges of temperature, with the result that large concentrations of aromatics are not obtained in any fraction. On the other hand, paraffinic and naphthenic hydrocarbons distil at much closer intervals.

A maximum concentration of aromatic hydrocarbons is usually found in fractions boiling a few degrees below the pure aromatic hydrocarbons.

The fact that aromatic hydrocarbons are distributed over a wide range of temperature results, likewise, in a disturbing effect on distillation of the saturated hydrocarbons which are partly carried away by fractions having nearly the same boiling point. This fact clearly follows from the distillation curves, which in the case of gasolines containing aromatics show less pronounced minima than in the case of aromatic-free gasolines.

For this reason we abandoned the distillation of gasolines containing aromatics, with a view to distilling in future only aromatic-free gasolines, as it is much more precise.

The proportion of heptane and methyl*cyclo*hexane in the Bucşani gasoline is very typical, as illustrating the reciprocal influence exerted by paraffins and naphthenes with close boiling points. Both hydrocarbons which occur in almost equal quantities distil mostly at an interval of 3° C., from 97° to 100° C. The distillation curve shows two maxima: one at 97–98°, corresponding to a fraction rich in *n*-heptane, and another at 99–100° C. rich in methyl*cyclo*hexane, and containing, likewise, *n*-heptane.

This example will suffice to show, therefore, the separation limit of our

column. Effective separation can be achieved only by redistilling the fractions, which would necessitate, however, much larger quantities of gasoline. Since, however, our object is to obtain as uniform fractions as possible, and not the separation of individual compounds, in order to enable the application of the analytical methods used, the efficiency of our column is quite sufficient.

In order to estimate the percentage of paraffinic and naphthenic hydrocarbons, we have adopted as basis the intervals shown in Table V, established on the basis of the maxima and minima of the distillation curve and the boiling points of the pure hydrocarbons. It is to be noted that in the case of all distilled gasolines the maxima and minima of the curves meet at the same points: $\pm$ 1° C. It is thus possible to determine the intervals for the various hydrocarbons as in Table V, since each interval comprises a maximum accompanied by clearly discernible minima.

TABLE V.

Boiling Intervals.	Nature of Hydrocarbon.
Paraffins	
50–65	*iso*Hexanes(2- and 3-methylpentane).
65–75	*n*-Hexane.
75–95	*iso*Heptanes(2- and 3-methylhexanes).
95–103	*n*-Heptane.
103–120	*iso*Octanes (a dimethylhexane in fract. 103–110° C. and 2- or 3-methylheptane in fract. 117–120° C.).
120–128	*n*-Octane.
128–145	*iso*Nonanes.
145–150	*n*-Nonanes.
Naphthene	
50–57	*cyclo*Pentane.
65–75	Methyl*cyclo*pentane.
75–85	*cyclo*Hexane.
85–95	Dimethyl*cyclo*pentanes.
95–104	Methyl*cyclo*hexane.
104–113	Naphthene non-identified (ethyl*cyclo*pentane, trimethyl*cyclo*pentanes).
113–126	Dimethyl*cyclo*hexanes.
126–133	Ethyl*cyclo*hexane and a nononaphthene with a 5-carbon cycle.
133–145	Nononaphthene in which trimethyl*cyclo*hexanes predominate.

Table V was established on the basis of the above considerations, and gives the composition of the respective gasolines. The figures in brackets corresponding to the original benzines represent the percentage of hydrocarbons of the respective class calculated in relation to the bulk of saturated hydrocarbons contained in the gasoline. These figures are, therefore, comparable with those found in fractions derived from aromatic-free gasolines.*

* It will be seen that in the case of the Bucşani gasoline, which was distilled, both in the aromatic-free state as well as with the aromatics, it contained the figures obtained for paraffins, *iso*paraffins and naphthenes show differences ranging from 1 to 2 per cent. Most of these differences are doubtless due to systematic errors during the estimation of aromatics.

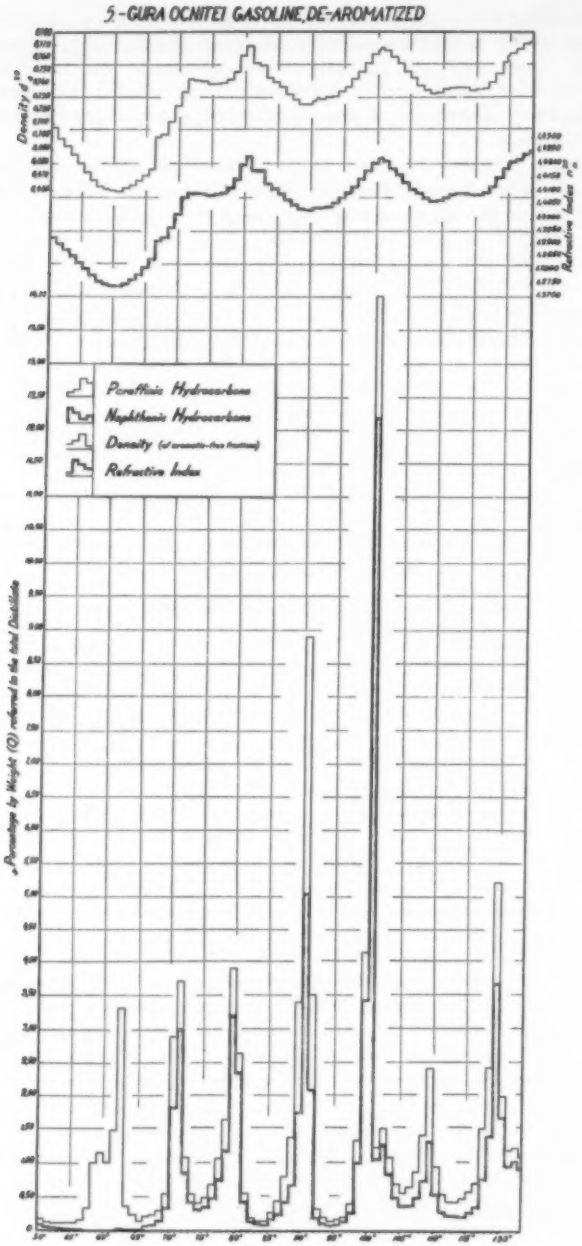

5.-GURA OCNITEI GASOLINE, DE-AROMATIZED
Density d 15°
Refractive Index n° D
Paraffinic Hydrocarbons
Naphthenic Hydrocarbons
Density (of aromatic-free fraction)
Refractive Index
Percentage by Weight (%) referred to the total Distillate

FIG. 5.

TABLE VI.

Composition of Fractions 50–150° C. of Gasoline Derived from :

	Merişor, O.N. = 63.	Bucşani, O.N. = 51·5.	Aromatic-free Bucşani, O.N. = 48.	Aromatic-free * Gura-Ocniţei, O.N. = 75.
n-Paraffins :				
n-Hexane . . .	5·4	5·3	6·5	3·0
n-Heptane . . .	4·8	8·3	8·8	3·1
n-Octane . . .	3·2	3·2	8·1	—
n-Nonane (−150°) . .	1·2	1·2	6·0	—
Total . . .	14·6 (15·8)	23·9 (31·2)	29·4	6·1
*iso*Paraffins :				
*iso*Hexanes . . .	5·4	5·1	5·7	9·4
*iso*Heptanes . . .	6·9	7·7	8·6	10·9
*iso*Octanes . . .	6·4	9·1	10·3	9·6
*iso*Nonanes . . .	5·2	8·4	9·2	—
Total . . .	23·9 (25·9)	30·3 (34·4)	33·8	29·9
Naphthenes :				
*cyclo*Pentane . . .	0·4	0·1	0·1	0·2
Methyl*cyclo*pentane . .	3·7	2·0	2·4	7·1
*cyclo*Hexane . . .	3·5	2·7	3·6	9·1
Dimethyl*cyclo*pentane .	6·0	2·7	3·4	10·8
Methyl*cyclo*hexane . .	9·3	7·3	9·2	20·4
Naphthene fract. 104–113° C.	3·6	1·2	1·5	4·9
Dimethyl*cyclo*hexane .	11·0	5·7	7·0	11·6
Naphthene fract. 126–150° C.	16·1	8·4	9·6	—
Total . . .	53·6 (58·2)	30·1 (34·3)	36·8	64·1
Aromatics :				
Benzene	1·7	2·0	—	—
Toluene	2·1	3·6	—	—
Ethylbenzene. Xylene .	4·1	6·5	—	—
Total . . .	7·9	12·1	—	—

* 7·5% aromatic hydrocarbons—Gura-Ocniţei gasoline.

We see, therefore, that the percentage of *iso*paraffins shows a variation of ± 5 per cent. and amounts to approximately 30 per cent. The percentage of naphthenes shows large variations. Whilst the proportion of naphthenes is about 60 per cent. for the two asphaltic gasolines, it is only 37 per cent. in the case of the gasoline of paraffinic origin.

The disturbing influence of normal paraffins on the octane number is clearly evident : whereas gasolines with a high O.N. contain only 6 per cent. of *n*-paraffins, paraffinous gasolines (with O.N. = 48) contain 30 per cent. of *n*-paraffins. The wide difference of the O.N. for the Merişor and Gura Ocniţei gasolines is solely due to a comparatively small difference (8 per cent.) in the contents of *n*-paraffins. The proportion of *iso*- and normal paraffins in Bucşani gasoline is roughly equal to 1 (33·8 : 29·4); in the Merişor gasoline it is 1·6 (25·9 : 15·8) and in the high-octane Gura Ocniţei gasoline it is 5 (29·9 : 6·1).

With regard to the nature of *iso*paraffins, examination of the distillation curves will show that in gasoline the predominating derivatives are those with a single side chain (methyl) and, namely, 2- or 3-methylhexane, or a mixture of both 2 : 3- or 4-methylheptanes, or a mixture of them, etc. The percentage of *iso*heptane together with dimethyl*cyclo*pentane in all fractions from 89 to 91 is considerable in all gasolines. The proportion of *iso*paraffins with several side-chains is smaller. Such compounds are found in fractions which boil at approximately 80° C. (an *iso*heptane), 107° C. (*iso*-octane) and 135° C. (*iso*nonane). No hypothesis is put forward here as to their structure.

Naphthenes are found throughout the 50–150° C. interval, with the exception of the range from 55° to 66° C., which is free from naphthenes in all the gasolines investigated. A small proportion of *cyclo*pentane is found in all gasolines. Methyl*cyclo*pentane, *cyclo*hexane, methyl*cyclo*hexane and dimethyl*cyclo*hexane are also surely found, as shown in the diagrams. The last two naphthenes are, quantitatively speaking, the most important hydrocarbons in gasoline. Methyl*cyclo*hexane constitutes 20 per cent. of Gura Ocniţei gasoline (fraction 50–123·5) and about 10 per cent. of the other gasolines.

In conclusion, it may be said that the three gasolines investigated in our laboratories possess very similar distillation curves. The maxima and minima of the curves are found at the same points. The gasolines differ only in the percentage of the various components contained in the mixture. In the paraffin series normal hydrocarbons predominate, or those with a single side-chain; in most cases the side-chain is probably composed of methyl groups. Paraffins with several side-chains or with complicated ramifications exist only in unimportant concentrations. In the naphthenic series up to the mononaphthenes, all possible homologues and isomers are represented. Among these, derivatives of *cyclo*hexane are contained in larger proportions than those of *cyclo*pentane.

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THE RAPID DETERMINATION OF SOLUBLE BITUMEN IN ROAD CARPETS.

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Summary.

A detailed account is given of two rapid but reliable methods employed by the author for the determination of soluble bitumen in road carpets.

The first consists of a hot extraction process in which a tall metal beaker replaces the usual Soxhlet form of apparatus. The sample is placed in a wire gauze basket suspended inside the beaker and loss of solvent is prevented by closing the top of the beaker with a condenser made from lead compo' tubing. The bitumen is determined by difference.

In the other method, a known volume of solvent is added to the sample and the mixture agitated until the bitumen has completely dissolved. An aliquot portion of the solution is then evaporated to dryness and the bitumen determined by direct weighing.

The necessary precautions and manipulative technique are described in detail in each case. Both methods are extremely simple in character but give results in good agreement with the British Standard method.

A method of determining by wet sieve analysis the proportion of material passing 200 British Standard mesh in the recovered mineral aggregate is also described.

Few chemists engaged in the asphalt industry have not experienced the need for a simple rapid and reliable method for determining soluble bitumen in road carpets, and particularly is this the case with those employed in the laboratory supervision of road constructional works. The procedure recommended by D. M. Wilson,[1] and adopted by the British Standards Institution,[2] is often unsuitable where results are urgently required, owing to the length of time necessary for the filtration. Asphalt cements containing large amounts of Trinidad Lake Asphalt and carpets incorporating these cements are notable in this respect. The difficulty is further accentuated where the size of the coarse aggregate necessitates the use of a large sample. For certain routine purposes, it may be possible to dispense with the determination of the percentage and sieve sizes of the coarse aggregate, and save time by taking comparatively small samples, but the procedure is obviously inapplicable where the sieve analysis of the recovered mineral aggregate is required.

It is not surprising, therefore, that many rapid methods of determining the soluble bitumen have been developed. In some, no attempt has been made to secure a very high degree of accuracy and the object has been simply to detect the major discrepancies from specification and enable appropriate remedial methods to be applied without loss of time. Nevertheless, a number of rapid methods is available, which can be relied upon to give results in close agreement with the standard method; these fall into two main classes:

(i) Hot extraction methods utilizing solvents such as trichlorethylene, perchlorethylene and benzene, in which the bitumen is usually found by difference.

(ii) Cold extraction methods with similar solvents, but employing a direct bitumen determination.

(i) Hot Extraction Methods.

Apparatus and Method for Hot Extaction of Bituminous Aggregates is described by Hubbard in Laboratory Manual for Bituminous Aggregates, 1916. This method, using the New York Testing Laboratory Extractor, has been widely used and consists of a cylindrical brass vessel for holding the solvent, inside which is suspended a cylindrical wire basket of 80 mesh wire cloth; an inverted conical condenser is fitted in the top so as to ensure that the vapours are condensed and percolate through the bituminous aggregate contained in the wire basket.

A well-known method belonging to this group is that described by D. M. Wilson,[3] which embodies a filtration under reduced pressure through a Kieselguhr filter cylinder of the Berkefeld type. The analysis may be completed comfortably in a working day and is suitable for the simultaneous extraction of a number of samples of road carpet. It does not appear to have been extensively adopted, probably because many works laboratories do not possess the requisite compressed air and vacuum lines.

A number of other forms of hot extraction apparatus are based on the principle of the Soxhlet extractor. The filter thimble is frequently replaced by a wire-mesh basket to avoid difficulties in adjusting the rate of refluxing to suit filtration. With the type of road carpets laid in this country, it has been found that if the filter paper is close enough to retain the fine mineral matter, the rate of filtration is usually slower than the rate of refluxing, with the consequence that fine mineral matter is liable to be washed over the top of the paper. Whatever type of apparatus is used, it is desirable that the dimensions of the extractor should be large enough to take samples weighing 1 to 2 kgms. A Soxhlet extractor of this size is necessarily an expensive item of equipment and is unlikely to find favour in laboratories where numerous soluble bitumen determinations are carried out.

The extractor developed at these laboratories (Fig. 1) described below does not suffer from this objection. It may be easily and cheaply constructed and is suitable for all grades of stone-filled asphalt. For mastic asphalt, a slightly modified form, described in a later section, is recommended.

Extractor for Stone-filled Asphalt.

The extractor for stone-filled asphalt consists of an aluminium or stainless steel beaker, 6 ins. diameter and 12 ins. high, inside which is suspended a cylindrical gauze basket, 5½ ins. diameter and 6 ins. high. Loss of solvent during extraction is prevented by placing on the top of the beaker a condenser made from a spiral of lead compo' tubing.

The basket may be easily constructed from a cylindrical tin 5½ ins. diameter with a press-on lid as follows:—three circular holes 1½ ins. diameter are cut in both lid and base and 85 B.S. wire gauze soldered over the holes. The major part of the cylindrical portion of the tin is then cut away leaving only a ½-in. rim attached to top and bottom. These two pieces are joined together with four brass strips ½ in. wide and 6 ins. long and the basket

completed by soldering 85 B.S. wire gauze around the outside of the framework. If desired, the top portions of two tins may be employed and a reversible basket made with a lid at the top and bottom. The basket is suspended by a wire passing around the rim of the beaker and attached to two hooks on the top of the basket.

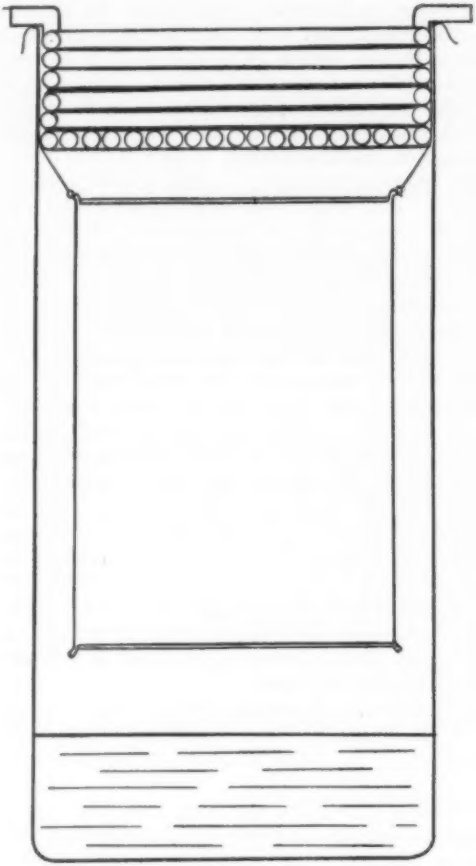

FIG. 1.

The condenser is made from lead compo' tubing $\frac{1}{4}$ in. external diameter wound in the form of a spiral and wired to a disc of perforated zinc. The spiral is made to fit inside the beaker and is provided with a few extra coils on the outside rim to give it the shape of a shallow trough.

Method for Stone-filled Asphalt.

A representative sample weighing at least 1 kgm. is obtained by the usual quartering procedure and dried in an oven at 105° C. The sample is then

introduced into the weighed basket. Approximately 500 ml. of trichlorethylene are transferred to the beaker and the basket suspended by wire about 1 in. above the surface of the liquid. The condenser is placed in position and the solvent maintained in a state of steady ebullition for 4 to 5 hours, after which time the apparatus is allowed to cool and the contents of the basket tipped out on to a shallow tray and placed in a warm position until the odour of solvent has disappeared. The basket and mineral aggregate are then dried in an oven at 105° C. for 1 hour and weighed.

The solvent contained in the beaker is carefully decanted into a 1 litre graduated flask and the mineral residue washed several times with trichlorethylene allowing 5 to 10 minutes before decanting each portion of wash liquid into the litre flask. The beaker containing the residual mineral matter is then dried and weighed; alternatively, the mineral matter may be transferred with solvent to a smaller receptacle.

The bitumen solution is diluted with solvent to exactly 1 litre and well shaken. An aliquot portion of 100 ml. is immediately measured into a graduated flask and transferred together with rinsings to a weighed silica dish. The solvent is evaporated as far as possible on a water bath, after which the dish is placed over a low bunsen flame and the temperature gradually raised until all carbonaceous matter has been removed. Precautions are taken during this process to prevent the bitumen from catching fire.

Finally, the residue is recarbonated and the weight of ash multiplied by the appropriate factor. The quantity of ash recovered by this process does not exceed 1 per cent. of the sample and for the purpose of computing the sieve analysis of the mineral aggregate it may be assumed to consist entirely of material passing the 200 mesh B.S. sieve.

The weight of bitumen in the sample is obtained by subtracting the total recovered aggregate from the weight originally taken for analysis.

A centrifuge may be employed if desired to separate the mineral matter from an aliquot portion of the bitumen solution. Such separations are, however, rarely complete, and the precaution of determining the ash content of the centrifuged aliquot should not be neglected. The centrifuge method is recommended when the sample contains organic matter insoluble in the usual solvents for bitumen, as is the case when certain native asphalts are present or mineral fillers which undergo considerable change in weight on ignition and recarbonation. The error arising from these causes is, however, comparatively small, and the soluble bitumen figure is unlikely to be more than 0·3 per cent. in error, even when the centrifuge process is not employed.

Extractor for Mastic Asphalt.

A basket made from 200 B.S. wire gauze is employed for mastic asphalt. It is constructed in a similar manner to the basket used for rolled asphalt, except that the interior of the basket is divided into four compartments by means of gauze discs supported on a central brass rod. The discs may be constructed from tin lids by cutting out 3 or 4 circles or sectors and covering the holes with wire gauze. The central brass rod is made in 4 tapped and screwed sections, each 1½ ins. long. A somewhat similar form of basket has been employed at the Road Research Station, Harmondsworth, Middlesex with very successful results.

The sample is warmed and pulled apart into pieces as small as possible and introduced into the basket as follows :—the first section is screwed to the bottom of the basket and approximately quarter of the sample added. The first disc and second section of rod are fitted into position and another portion of sample transferred to the basket. The process is repeated with the second and third discs and subsequent procedure is the same as that described for rolled asphalt, except that a longer period of extraction is usually necessary. If circumstances permit, the extraction may be allowed to proceed overnight.

The foregoing methods give results in good agreement with those for the standard procedure described in B.S.S. 598.[4]

Some typical figures obtained with samples of steam-rolled and mastic asphalts are shown in Table I.

TABLE I.

Sample.	A.C.	Soluble bitumen.	
		Hot extraction, per cent.	B.S.S. 598, per cent.
Steam-rolled asphalt	Fluxed epure	8·8	8·5
,, ,, ,,	Asphaltic bitumen	7·4	7·2
,, ,, ,,	,, ,,	7·7	7·5
Mastic asphalt	,, ,,	12·7	12·7
,, ,,	Fluxed epure	12·0	11·8
,, ,,	Asphaltic bitumen	9·4	9·4
,, ,,	,, ,,	12·7	12·7
,, ,,	,, ,,	11·0	11·2

(ii) COLD EXTRACTION METHODS.

A feature of the majority of these methods is that a known volume or weight of solvent is added to the weighed sample and after taking suitable precautions to secure complete solution of the bitumen and the removal of the bulk of the mineral matter, an aliquot portion of the solution is evaporated to dryness. The automatic burette method described by D. C. Broome,[5] and designed for use with mastic and rock asphalts follows these lines, as does that put forward by I. Hvidberg.[6] The method described below is based on similar principles, but incorporates certain modifications which give improved accuracy and renders the method of more general utility. The method is suitable for all types of bituminous road carpets and can be recommended for routine control purposes. It is extremely simple, requires no elaborate apparatus and can be completed in approximately 3 hours.

Method. (*Applicable to B.S.S. Nos.* 347, 348, 594, 595, 596 & 597.)

The sample is warmed in an oven until soft and, if present, precoated chippings are removed and discarded. The sample is then pulled apart into pieces as small as possible and quartered to the approximate weight given in Table II. A sampling procedure is employed and no attempt is made to adjust the weight of the sample to the exact figure shown in the table.

TABLE II.

Minimum Weight of Sample.

Mastic asphalt, ungritted	100 gms.
Mastic asphalt containing aggregate passing ¼ in.	100 ,,
Mastic asphalt containing aggregate ¼–¾ in.	250 ,,
Sand carpet	100 ,,
Rolled asphalt containing less than 25 per cent. ¾ in. stone	500 ,,
Rolled asphalt containing more than 25 per cent. ¾ in. stone	1000 ,,
Rolled asphalt, binder course	1000 ,,
Compressed rock asphalt	100 ,,

The sample is dried in an oven at 105° C. and weighed. It is then placed in a glass bottle or metal drum and a measured quantity of solvent (Table III) added from a graduated flask, allowing 30 seconds for the flask to drain. The solvent may be carbon disulphide, chloroform, perchlorethylene or trichlorethylene.

TABLE III.

Sample weight.	Volume of solvent.
100 gms.	1000 ml.
250 ,,	2000 ,,
500 ,,	3000 ,,
1000 ,,	5000 ,,

The temperature of the solution is read, after which the container is vigorously shaken for 30 seconds and then set aside and re-shaken four more times at quarter-hourly intervals; finally the solution is allowed to remain undisturbed for a further quarter of an hour.

Approximately 50 ml. of bitumen solution are removed and centrifuged for 2 minutes at 2000 to 4000 r.p.m., the average radius of the path of the liquid in the centrifuge tube being 4½″. The centrifuge tubes are provided with tin-foil caps to minimize loss of solvent by evaporation. A portion of the centrifuged liquid is decanted into a beaker and the temperature quickly adjusted to its former value. 10 ml. of solvent are then transferred with a pipette (calibrated for use with the particular solvent) into a weighed dish. The solvent is evaporated on a water bath and the residue dried at 160–170° C. for half an hour, or at 105° C. for one hour and weighed. The residue is ignited and the ash recarbonated. In calculating the soluble bitumen content, an allowance is made for the fact that the volume of bitumen solution is always slightly greater than the volume of solvent.

If A = weight of bitumen in aliquot,
B = weight of sample,
C = volume of aliquot,
D = total volume of solvent,
E = weight of ash in aliquot,

then the volume of bitumen solution $= D\left(1 + \frac{A}{C}\right)$

and percentage soluble bitumen $= \frac{100\,AD}{BC}\left(1 + \frac{A}{C}\right)$

The percentage ash given by $\frac{100\,ED}{BC}$ should not exceed 1 per cent.

If a centrifuge is not available, the container after shaking in the manner described above may be allowed to stand overnight and 10 ml. of the solution withdrawn without disturbing the mineral matter at the bottom of the container. Should the ash exceed 1 per cent., another 10 ml. aliquot is diluted to 150–200 ml. and filtered through a No. 5 Whatman, 11-cm. filter paper. The filtrate and washings are then transferred to a weighed dish and evaporated to dryness and ashed. The extra time involved in this procedure amounts to approximately 2 hours. The same method may be used in conjunction with a centrifuge process if so desired.

TABLE IV.

Sample.	A.C.	Soluble bitumen.	
		Cold extraction, per cent.	B.S.S. 598, per cent.
Mastic asphalt	Asphaltic bitumen	10·0	9·9
,, ,,	,, ,,	9·4	9·2
,, ,,	,, ,,	9·3	9·1
,, ,,	,, ,,	9·4	9·4
Steam-rolled asphalt	,, ,,	9·4	9·2
Mastic asphalt	,, ,,	15·7	15·6
,, ,,	,, ,,	14·0	13·9
,, ,,	,, ,,	15·4	15·5
,, ,,	Fluxed epure	15·2	15·0
,, ,,	,, ,,	16·0	15·7

The results given in Table IV were obtained using trichlorethylene as a solvent. The figures quoted are in close agreement with those obtained by the standard method.

When a sieve analysis of the mineral aggregate is required, the bitumen solution remaining after the determination of soluble bitumen is agitated and passed through an 8- and a 200-mesh B.S. sieve, fitted together over an 8-in. funnel. The residual mineral matter is washed several times with trichlorethylene and finally transferred to the 8-mesh B.S. sieve. Fine particles of filler adhering to the coarse aggregate are removed with a jet of trichlorethylene from a wash-bottle fitted with a blow-ball. The aggregate retained on the 8-mesh B.S. sieve is then dried, weighed and sieved in the usual manner. It is not assumed to consist entirely of plus 8 material, but is re-sieved on the 8-mesh B.S. sieve.

The fine material on the 200-mesh B.S. sieve is washed three or four times with trichlorethylene, then once with alcohol (95 per cent. I.M.S.) and finally with a stream of tap water until the −200 material has been removed. This point is ascertained by examining the water passing through the sieve. It is an advantage to employ a metal spiral in which a number of small holes have been bored to distribute the water over the entire surface of the sieve, as by this means sieving may be carried out with practically no attention or effort on the part of the operator. When the washing process has been completed, the residue is rinsed with alcohol, dried and weighed. The proportion of material passing 200 mesh is obtained by subtracting the weight of aggregate retained on the 8- and 200-mesh sieves

from the total weight of mineral aggregate. The aggregate retained on the 200-mesh sieve is dry-sieved in the usual manner.

Wet sieving possesses pronounced advantages over the more usual dry sieving and gives more accurate and consistent results. It is to be hoped that in course of time it will entirely replace the older method. The proportion of —200 material found by dry sieving (B.S.S. 598) is invariably low, as filler adheres to the coarse aggregate and is lost when the latter is separated on the 8-mesh sieve. The error so introduced depends both on the quantity and nature of the coarse aggregate and is frequently very marked. For many years now, it has been the custom to dry sieve mineral aggregates and fillers, but the process has little to recommend it. The separation of the —200 fraction by wet sieving not only ensures correct and consistent results, but eliminates an extremely tedious and dusty operation and reduces the wear and tear on the 200-mesh sieve. It can be employed in the presence of a Portland cement filler and is particularly useful for soft limestone aggregates which are difficult to deal with by the normal method.

In the course of investigating the cold extraction method, attention was directed to certain possible sources of error and a number of experiments were carried out with a view to checking the accuracy of the method and improving the general manipulative technique. These experiments may be grouped under the following headings :—

(*a*) Correction for solution volume of bitumen.
(*b*) Correction for volatilisation of solvent during the centrifuge process.
(*c*) Calibration of measuring vessels.
(*d*) Procedure for drying residue.
(*e*) Ashing procedure.
(*f*) Check determinations.

(a) *Correction for Solution Volume of Bitumen.*

Weighed amounts of bitumen were added to measured volumes of solvent and the increase in volume noted. It was apparent from the results that for the purpose of calculating the correction it would be sufficiently accurate to regard the increase in volume as equal to the volume of bitumen and to take the density of the bitumen as unity. Thus, employing the nomenclature given above, it could be assumed that the volume of bitumen solution would be greater than the volume of solvent by $\frac{AD}{C}$.

(b) *Correction for Volatilisation of Solvent during the Centrifuge Process.*

A centrifuge tube approximately 2 cms. diameter and 35 ml. capacity with a hemispherical end was employed in these experiments and was weighed before and after centrifuging. The percentage loss of solvent when the tubes were filled to capacity was found to be somewhat greater than when the tubes were partly filled, but by placing tin-foil caps over the open ends of the tubes, the loss in all cases could be reduced to insignificant proportions. The caps were made by placing a circle of tin-foil about 2 cms. diameter over the top of the tube and smoothing the projecting portion into contact with the outside of the tube. The efficacy of this device is clearly indicated by the figures given in Table V.

TABLE V.

Solvent.	Loss after centrifuging 2 mins.		Room temp., ° C.
	With. cap. per cent.	Without cap, per cent.	
Carbon disulphide . .	0·06	0·60	28·8
Trichlorethylene . . .	0·02	0·27	,,
Chloroform	0·03	0·33	,,
Carbon tetrachloride . .	0·02	0·28	,,
Perchlorethylene . . .	0·003	0·11	,,

(c) *Calibration of Measuring Vessels.*

Pipettes. It is usual to calibrate pipettes with distilled water. For the present work, however, it was considered preferable to calibrate with the particular liquid to be employed and ascertain by direct weighing the ratio of the weight of solvent delivered by the pipette to the weight delivered by the measuring flask.

Measuring Flask. A 1-litre measuring flask filled to the mark with trichlorethylene was found to deliver 999·0 ml. when a drainage time of 30 seconds was allowed. The error in measuring the requisite volume of solvent could, therefore, be assumed to be negligible.

(d) *Procedure for Drying Residue.*

The following experiments were carried out with a view to ascertaining whether loss of volatile hydrocarbons occasioned by drying at 160–170° C. introduced an error into the analysis. 0·1 gm. of several grades of bitumen and flux oil were transferred to porcelain dishes, dissolved in 10 ml. trichlorethylene and the solutions evaporated to dryness on a water bath. The residues were dried either at 105° C. or at 160–170° C. and reweighed. The results shown in Table VI suggest that in the absence of volatile flux oils or soft bitumens a drying time of half-an-hour at 160–170° C. will give satisfactory results. In case of doubt a drying time of one hour at 105° C. is recommended.

TABLE VI.

Bitumen.	Drying period.		Change in weight, mgms.
	105° C., hrs.	160–170° C., hrs.	
Mexphalte 65 . . .	2	—	+ 1·0
,, . . .	—	1	Nil
,, . . .	—	4	− 2·0
Mexphalte 80/90 . . .	—	½	− 0·4
Texaco E flux oil . . .	—	½	− 1·6
Shell flux oil . . .	2	—	− 2·9
,, ,, ,, . . .	—	½	− 15·8
16% Shell flux oil, 84% epure	1	—	Nil
,, ,, ,, ,, ,,	2	—	− 0·6
,, ,, ,, ,, ,,	3	—	− 1·3
,, ,, ,, ,, ,,	4	—	− 1·3
,, ,, ,, ,, ,,	—	½	− 5·2

(e) *Ashing Procedure.*

A porcelain dish may lose in weight on strong ignition and for this reason it is advisable not to heat the residue more than is necessary to effect removal of carbonaceous matter. The recarbonation process is carried out as follows :—

The ash is allowed to cool and moistened with a few drops of ammonium carbonate solution. The excess is evaporated on a steam bath and the dish then gently warmed until the smell of ammonia is no longer perceptible. The dish is then re-weighed.

(f) *Check Determinations.*

10 gms. of Mexphalte 30/40 were placed in a flask and 1000 ml. solvent and 100 gms. limestone powder added. After shaking and adjusting the temperature of the solution as prescribed in the method, a 10 ml. aliquot was evaporated to dryness, dried half-an-hour at 160–170° C., weighed, ashed and the residue recarbonated. Similar experiments were carried out adding pulverized silica and Portland cement. In all cases the weight of bitumen obtained was slightly less than the theoretical amount, presumably on account of absorption of bitumen by the mineral aggregate. The actual figures are shown in Table VII.

TABLE VII.

Solvent.	Aggregate.	Wt. of bitumen in aliquot.
Carbon disulphide	Limestone	0·0996
Chloroform	,,	0·0994
Carbon tetrachloride	,,	0·0981
Perchlorethylene	,,	0·0985
Trichlorethylene	,,	0·0986
,,	Pulverized silica	0·0988
,,	,, ,,	0·0985
,,	Portland cement	0·0978

In conclusion, the author wishes to express his thanks to the Director of these laboratories, Mr. H. B. Milner, M.A., for permission to publish and to record his appreciation of the efficient manner in which Mr. A. H. Clarke performed most of the analytical work.

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References.

[1] D. M. Wilson, *J.S.C.I.*, 1931, **50**, (28), 599–600.
[2] British Standards Institution, B.S.S. 598, 1936, "Methods for the Sampling and Examination of Bituminous Road Mixtures."
[3] D. M. Wilson, *J.S.C.I.*, 1933, **52**, (28), 597–81.
[4] British Standards Institution, B.S.S. 598, 1936, "Methods for the Sampling and Examination of Bituminous Road Mixtures."
[5] D. C. Broome, "The Testing of Bituminous Road Mixtures," Arnold, London (1934).
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THE INSTITUTE OF PETROLEUM.

SPECIAL GENERAL MEETING.

A Special General Meeting of the Institute was held at the Royal Society of Arts, John Street, London, W.C.2, on Tuesday, 10th January, 1939, at 5.30 p.m., for the purpose of considering and, if thought fit, passing with or without amendment certain Temporary Regulations relating to the Transfer of Members, Associate Members and Associates.

THE PRESIDENT, LIEUT.-COL S. J. M. AULD, O.B.E., M.C., D.Sc., occupied the Chair.

THE SECRETARY (MR. S. J. ASTBURY) read the proposed Temporary Regulations, as under :

TEMPORARY REGULATIONS RELATING TO THE TRANSFER OF MEMBERS, ASSOCIATE MEMBERS, AND ASSOCIATES.

(*In force up to 30th June*, 1939.)

(*a*) All Members, Associate Members, and Associates of the Institute of Petroleum, whose names were on the register as such on the 31st December, 1938, shall be entitled to apply for transfer to one of the classes of Fellows, Members, or Associate Members in accordance with the Regulations given hereunder.

(*b*) As soon as possible after the Special General Meeting at which these Temporary Regulations are approved by the members, and in any case before 30th June, 1939, any Member, Associate Member, or Associate desirous of being transferred to another class of membership should indicate to the Council on a Form prescribed for the purpose the class of membership to which he applies to be transferred.

(*c*) No transfer shall become effective before the 1st January, 1939, and no transfer shall be deemed valid until the appropriate Transfer Fee, if any, shall have been paid.

MEMBERS.

(*d*) A Member of the Institute of Petroleum whose name was on the register as such on the 31st December, 1938, is entitled to apply for transfer to the class of Fellow, and the decision of the Council on his application shall be final. No Transfer Fee shall be required.

ASSOCIATE MEMBERS.

(*e*) An Associate Member of the Institute of Petroleum is entitled to apply for Transfer to the class of Fellow or Member, and his application for transfer shall be submitted to and decided upon by the Council, whose decision shall be final; or he may continue an Associate Member of the Institute.

A Transfer Fee of £1 1*s*. shall become payable on transference of an Associate Member to the class of Fellow or Member.

ASSOCIATES.

(*f*) An Associate of the Institute of Petroleum is entitled to apply for transfer to one of the classes of Fellow, Member, or Associate Member, and his application for transfer shall be submitted to and decided upon by the Council, whose decision shall be final.

No " Associate " may continue as such after the 30th June, 1939.

On transference to the class of Fellow or Member an Associate shall pay a Transfer Fee of £1 1*s*. On transference to the class of Associate Member no Transfer Fee will be required.

PUBLICATION OF TRANSFERS.

(*g*) Notification of applications for transfer will not be published in the Journal, but all transfers will be announced in the Journal as and when they become effective.

The PRESIDENT, in proposing the adoption of the Regulations, said they had been prepared by the Council and were the result of a considerable amount of deliberation. He did not think they required any explanation from him, as they were quite clear.

MR. JACKSON seconded the motion.

MR. BUTTERFIELD said that, as a member of long standing, he would like to ask for a little explanation of the grounds of transfer of Members to the grade of Fellows. Up to the end of last year Members were in the highest category of membership of the Institute, but he gathered that in future the highest category would be that of Fellow. Would those who were Members at the end of last year be automatically transferred, on application, to the grade of Fellow, or was it intended that some discretion should be exercised in the matter? The question was raised, he thought, by the words in Regulation (*d*): "the decision of the Council on his application shall be final." The Council knew all those who were at present Members, and must be in a position to make up their minds whether, on application, those Members should be transferred *en bloc* to the grade of Fellow. If the Council did not intend to do that, but to exercise some discrimination, the grounds on which the applications would be considered should, he thought, be indicated to the Members. If it was not intended to exercise such discrimination, the words "the decision of the Council on his application shall be final" might well be omitted.

THE PRESIDENT said he would answer Mr. Butterfield's question in the same way as that in which he had answered it previously, by saying that the matter would be dealt with most generously by the Council, but the Council wished to have the power to discriminate if and when necessary. He did not like using the word "discriminate," because it implied something rather different from what the Council had in mind. He thought the fact that in the definition of Fellows it was stated that a Fellow must have "advanced the science and technology of petroleum" implied that there might be cases in which, if the standard of a Fellow was to be maintained, it would not be suitable to transfer a Member to the grade of Fellow without consideration. He thought also that the fact that the matter was given consideration should be welcomed by the Fellows. They would feel that they had not been elected by a stroke of the pen, but that it had been done carefully; there being first a definite, deliberate instruction from the Election Committee to the Council and then a reconsideration of the matter by the Council.

DR. J. A. L. HENDERSON asked whether the twenty-four Founders would continue to be life members of the Institute when transferred to the grade of Fellow.

THE PRESIDENT said that that would undoubtedly be the case.

If there were no other questions, he would put the resolution that the Temporary Regulations read by the Secretary be adopted.

The resolution was carried.

THE PRESIDENT said there was no other business and declared the meeting closed.

ERRATUM.

Physical and Chemical Constants of Normal Paraffins by D. J. W. KREULEN.

Journal, Vol. 24, No. 180, October 1938, p. 557. Table II last column. Values for Wp should be 0·95, 0·85, 0·77, 0·61, 0·30, 0·18, 0·13, 0·04 for molecular weights from 481 to 206.